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TEXTILE RESEARCH JOURNAL

VOL. XXVII, NO. 9 SEPTEMBER 1957

The Significance of Wool Fiber Crimp

Part I: A Study on the Worsted System[1]

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Abstract

Two batches of Merino wool, closely matched in fiber diameter and length, but differing in their crimp content, have been processed into worsted yarn on the French system and converted into fabric. The changes in the properties of the fibers through processing have been followed, and the properties of intermediate assemblies and of the fabric examined.

The fiber crimp was found to persist through processing and to exert an effect on the properties of top, roving, yarn, and fabric. The wool with greater crimp content produced twistless assemblies with greater cohesion; performed better in spinning; gave rise to a bulkier yarn with lower stiffness in extension and lower breaking extension; and resulted in a thicker fabric with softer handle, smoother appearance, lower resistance to air-flow, slightly lower bending stiffness and better drape, and slightly better recovery from extension and from wrinkling. Crimp appeared to have no influence on the tensile properties of the fabric, or on its resistance to flex abrasion.

Introduction

The importance of crimp as a characteristic affecting both processing behavior of fibers and the properties of textile assemblies is generally accepted, but little factual information on the subject is available. It is known, qualitatively, that crimp is retained through processing to a greater extent in wool fibers than in the thermoplastic synthetics; the only evaluation of the changes in wool fiber crimp configuration in processing is the work of Chaikin et al. [9], who investigated the changes in crimp (length contraction) through worsted yarn manufacture by the autoradiography of tracer-labeled fibers. Their results indicate some loss in crimp in topmaking, little further change in drawing, and the extension of the fibers by several per cent in spinning. The changes which occur through processing in the fiber mechanical properties associated with crimp have been investigated only in a preliminary manner [8, 19, 41], and the information available concerning the effect of fiber crimp on textile processing performance and on the properties of fiber assemblies is fragmentary [33, 43].

The subject of crimp is of significance to the wool grower and buyer as well as to the textile technologist. In the case of Merinos, the quality grading, performed by visual and tactile examination, is

[1] Paper presented in part at the Gordon Research Conference on Textiles, New London, N.H., July 13, 1956.

based primarily on the staple crimp and only secondarily on fiber fineness and other factors [27, 35, 47]. The reasons for this emphasis on crimp in the appraisal have not, to our knowledge, been elucidated. It may be that crimp is taken as an indication of diameter ("the confidence of leaders in the wool trade in the reliability of crimp as a guide to diameter is almost complete" [36]); or that there is simply a misjudgment of fiber diameter due to the optical effect which makes crimped fibers, singly or in mass, appear finer than crimpless ones (the possible relation between this and the trade belief cited above needs no belaboring); or that crimp is in fact considered to have a technological significance overriding that of fiber fineness. Staple "character"—the distinctness and evenness of the staple crimp—is regarded as an index of fiber diameter and length uniformity, and thus indirectly of quality; the association, which is probably intuitive, is erroneous [28].

While there is an over-all relationship between fiber fineness and staple crimp, the correlation is by no means perfect [27, 36]. In consequence, quite serious divergencies can occur between the expert appraisal of quality grade, derived primarily from the staple crimp, and a grade assignment based solely on the fiber diameter [2, 39]. It is thus of interest to determine whether wool "quality," in a broad sense, is satisfactorily defined in terms of fiber diameter alone, or whether a better estimate is furnished by the expert assessment.

The work reported in this paper is aimed at elucidating the effect of fiber crimp on the properties of worsted structures, and at establishing what changes in the fiber crimp configuration take place as a result of processing. This being the first investigation in the field, it was designed as a single-factor study. Two wools were selected, as alike as possible in all their shape parameters other than crimp, and manufactured into textile structures under closely similar processing conditions. The mechanical properties of these assemblies and the properties of their constituent fibers were examined; the relationship between the assembly and fiber properties was then studied. The effect of crimp on processing performance was not followed in detail.

Two matters are considered before the details of the processing investigation can be conveniently discussed:

a. The method of crimp measurement used is described, and some data are presented to show its validity.

b. The two wools selected for the study are characterized in respect of their physical, mechanical, and chemical properties.

Crimp Measurement

Staple crimp frequency is readily measurable directly; the amplitude of the crimp wave can also be determined, although with less precision [5]. The staple crimp is, however, not a completely reliable index of the fiber crimp. The fibers withdrawn from a staple exhibit a wide range of crimp configurations, and their mean crimp wave length does not

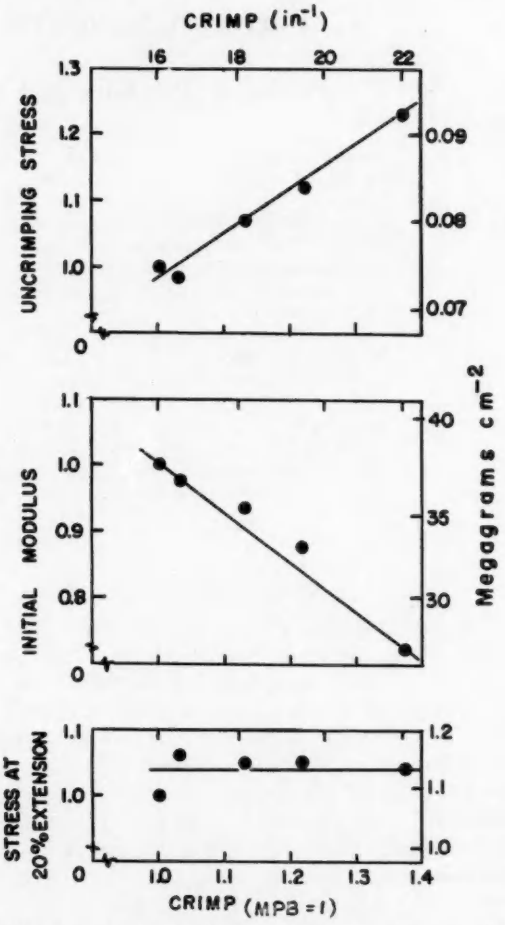

Fig. 1. The relationship between fiber tensile properties, tested at 65% R.H. 70° F., and the fiber crimp frequency, in five strains of Australian Merino Wool (MPB = 1).

necessarily coincide with that of the staple [21]. The staple crimp configuration is imposed on the fibers by external constraints; when released from these constraints, the fibers tend to assume their "natural" helical conformation, but can do so only when the set-in strains are removed by a treatment such as exposure to steam [46]. In the absence of such relaxation, the fibers assume a shape intermediate between the staple one and their natural one; the result is an irregular three-dimensional kinkiness. The geometry of such a configuration is difficult to measure and interpret [6], but attempts to do so indirectly have been made [*e.g.* 1].

In the present study, visual measurements of fiber crimp frequency were made on a projected image of a vertically suspended fiber at five-fold magnification. The fiber was fixed visually in a straight-but-crimped form, and the number of crimp waves was counted; an individual operator could obtain reasonably reproducible and consistent results. A tension approximating the "uncrimping force" (0.25 g.) was then applied at the lower end, and the length of the fiber measured. (N.B. The fiber is not completely straight under these conditions.) The crimp frequency was computed in terms of crimps per inch of extended length.

The main index of fiber crimp employed was not a geometric, but a mechanical one, namely the stress required to uncrimp the fiber, defined as the force per unit area developed at the intersection of the prolonged Hookean slope of the force-extension curve with the extension axis [18, 19]. Such a parameter is well defined and readily determined, but that is not the sole reason for its utilization; the stress is a measure of "the most important aspect of crimp content—its ability to perform against externally applied forces" [6]. In fact, it is impossible to separate the geometric and mechanical aspects of the crimp in the case of wool, since they are highly correlated. The relationship is illustrated by data obtained on fibers derived from five pure strains of Australian Merino wool of widely different crimp levels. Shoulder samples with a mean fiber diameter of 22μ were selected from five sheep of each strain; the fiber crimp frequency was measured as described above, and the fiber cross-sectional area was determined vibroscopically on 1-in. lengths of solvent-degreased and water-rinsed fiber [31]. The fibers were then extended on the Instron Tensile Tester through the Hookean region at 10%/min. rate of extension, and, after an overnight rest, to break at 50%/min. [19]. Four fibers per sheep (20 per strain of sheep) were tested dry (at 65% R.H., 70° F.), and the same number wet (in 0.1 *M* sodium borate, pH 9.2; see Appendix A) after an overnight soaking in the test solution. The results obtained are summarized in Table I.

In Figure 1, the uncrimping stress, Young's modulus, and stress at 20% extension, determined at 65% R.H., are plotted against the fiber crimp. The close relationship between the uncrimping stress and the crimp frequency is evident, as is the lowering effect of crimp on the modulus measured at low rates of strain [12, 18, 32],[2] and its negligible in-

[2] In the absence of crimp, the quasi-static modulus of Merino wool is closely similar to that of human hair. A sample of practically crimpless fibers from a mutant Merino sheep (kindly supplied by Dr. G. Laxer) gave values of 49.6 megagrams cm.$^{-2}$ dry, and 24.9 wet.

TABLE I. Crimp Level and Fiber Tensile Properties of Samples from Five Strains of Australian Merino Wool

Property	Test condition	Sheep strain: Non-peppin fine	Non-peppin medium	Non-peppin strong	Medium peppin A	Medium peppin B	95% Limits of ind. means	Within-strain C.V. % (avg.)
Staple crimp, in.$^{-1}$		17.6	10.2	7.2	9.2	7.8	±0.9	20
Fiber diameter, μ*		22.0	22.1	22.3	22.0	22.0		
Fiber crimp, in.$^{-1}$		21.9	19.4	18.1	16.4	16.0	±1.6	21
Fiber diameter, μ**		21.9	22.4	24.6	22.8	22.8	±1.1	15
Uncrimping stress,	Dry	0.092	0.084	0.080	0.074	0.075	±0.009	31
megagrams cm.$^{-2}$	Wet	0.030	0.028	0.026	0.026	0.023	±0.003	33
Young's modulus,	Dry	26.9	32.8	35.0	36.5	37.4	±2.3	15
megagrams cm.$^{-2}$	Wet	10.7	13.2	14.8	15.4	14.7	±1.2	19
Stress at 20% extension,	Dry	1.13	1.15	1.15	1.16	1.09	±0.05	8
megagrams cm.$^{-2}$	Wet	0.43	0.46	0.46	0.47	0.45	±0.02	7

* Bulk samples.
** Fibers used in stress-strain work.

fluence on properties beyond the Hookean region, exemplified by the stress at 20% extension.

The change in modulus with crimp is as great as that in uncrimping stress; the initial modulus might therefore be considered a valid mechanical index of the fiber crimp, particularly as it has the advantage of a lower variance. This possibility is limited to wool unmodified chemically or mechanically, as there is no way of differentiating between changes in modulus due to crimp and those due to other causes, such as chemical damage.

The uncrimping stress and the modulus show the same trend with crimp under both sets of conditions, but the dry test data exhibit somewhat less scatter. For this reason, and also because it was desired to measure the crimp content of fibers from various assemblies without prior relaxation of imposed strains, the dry uncrimping stress was used as the index of fiber crimp content throughout the processing study.[3]

Materials

Two Australian Merino wools, each originating from one commercial flock, were chosen for the study on the basis of preliminary staple crimp and fiber diameter measurements. The flock which yielded the "high crimp" wool was of a "fine Merino" strain grown on the northern plains of New South Wales. The other flock (low crimp), grown on the southern highlands of New South Wales, was of a "strong Merino" strain known as "South Australian." The selection of individual fleeces was made at shearing on visual estimates of staple crimp, fineness, and soundness. The crimp acceptance levels were: high crimp, not less than 13 crimps/in., low, not more than 10½; fleeces were rejected if they appeared very much finer or very much coarser than 62's, or if any indication of a "break" in the staple was present [34]. Eleven hundred pounds greasy of each type were thus obtained in the form of skirted fleeces. This material was used in the trials without further sorting.

Fiber Diameter and Quality Grade

The mean diameters of the two wools in the top stage, determined by the projection microscope ac-

[3] The uncrimping energy is probably a more direct index of the ability of the crimp to perform against external forces than the uncrimping stress. Unfortunately, the computation of the energy from Instron chart data is laborious. Evans and Montgomery [19] suggested the use of the product of uncrimping force and the uncrimping length as an approximation, and showed that its value is proportional to the energy for fibers withdrawn from top and yarn. In the present study, an equally good correlation was observed in raw wool, top, roving, and yarn. However, the relationship broke down in the case of fibers withdrawn from fabric, the product becoming much larger relative to the energy than in the earlier stages. This anomaly was due to a change in the shape of the stress-strain curve caused by the impression of fabric crimp onto the fibers. The use of the approximation was therefore abandoned. The limited measurements of energy which were made showed this parameter to behave similarly to the stress. Since the latter is more conveniently measured, it was adopted as the index of crimp.

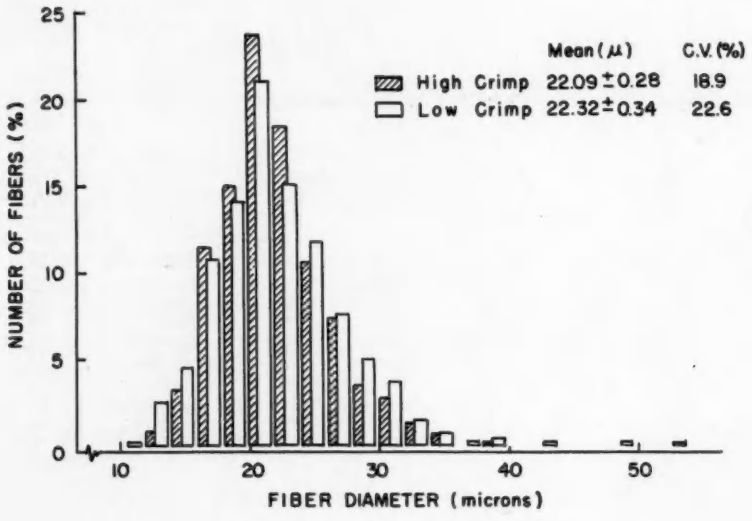

Fig. 2. Fiber diameter distributions in the top stage.

TABLE II. Fiber Fineness in the Top Stage and A.S.T.M. Quality Grade

	High crimp*	Low crimp*	64's Grade (A.S.T.M. D472–55T)
Average diameter, μ	22.1	22.3	21.1–22.5
Fibers, %			
10.0–30.0 μ	94.7	92.9	$\geqslant 92$
30.1 and over	5.3	7.1	$\leqslant 8$
40.1 and over	—	0.5	$\leqslant 1$

* 900 fibers.

cording to the I.W.T.O. method [4], are listed in Table II, together with the distributions in terms of the A.S.T.M. standard [2]. Both wools satisfy the A.S.T.M. specification for 64's grade. In contrast with this, expert assessment of the wools in the grease, by a buyer familiar with Australian wools, resulted in considerably different quality assignment for the two samples: the high crimp was described as 64's and the low crimp as 58's–60's.

The fiber diameter distributions of the two wools in the top are presented in histogram form in Figure 2. The data show that, while the mean diameters of the wools are indistinguishable ($P > 0.4$),[4] there is a real difference in the dispersions ($P < 0.001$), the low crimp wool having the greater spread and possessing, in trade parlance, a "coarse edge."

Part of the greater variation in the low-crimp wool is due to an along-the-staple component (Table III), evidently caused by fluctuations in nutrition during its growth period [34]. The wool is, nevertheless, sound in terms of the usual sorter's and buyer's criteria.

The imperfect match in the diameter distributions is of some concern, since certain fiber properties, particularly bending stiffness, are highly diameter-dependent; these in turn may control certain properties of the resultant fabric, such as its flexural rigidity and resistance to compression, and the more complex characteristics related to them, for example, handle and drape. Hence, the diameter effect could mask the influence of other fiber properties, particularly crimp in the present instance.

The possible magnitude of the diameter effect on assembly properties is evaluated in Appendix B.[5] It is shown that the sum of the fourth powers of the diameter in the same weight of fiber is 10.7% greater for the low-crimp than for the high-crimp wool. Consequently, a 10.7% difference between wools is to be expected in any fabric characteristic which is entirely controlled by a fiber property which varies with the fourth power of the diameter (such as bending stiffness). In practice, there is no doubt that every fabric property, even flexural rigidity, is influenced by a number of fiber characteristics, most of which have a lower diameter dependence than bending stiffness. The effect of the difference in fiber diameter distributions between the wools or the properties of fabrics made from them will, therefore, be smaller than 10.7%; no clear estimate of its magnitude can be attained in the present state of knowledge concerning fiber-fabric relationships.

Length and Crimp

The staple length and crimp, and the single fiber length in the raw state are reported in Table IV, together with the scouring yield, contour ratio (measured on Hardy cross sections), and the uncrimping ratio (extended fiber length/staple length). The latter was determined by measuring, on the single fiber tester [41], the length of fibers withdrawn from cut 1-in. portions of staple; the middle and base parts of the staple were used for this purpose.

Before discussing the properties of the wools further, it is desirable to indicate their crimp level in terms of the average crimp-diameter relationship. Duerden's scale, obtained from measurements on S. African wools [13], may be expressed in the form $n = 23000/(d^{\frac{1}{2}})$, where n is crimp frequency in crimps/in. and d the fiber diameter in microns [26]. For a diameter of 22μ this relationship gives 10.1 crimps/in.; conversely, a crimp level of 14.8 corresponds to a diameter of 18.8μ, and that of 8.9 to 23.5μ. If the more recent data of Lang [27], de-

TABLE III. Fiber Diameter (μ) at Different Levels in the Staple by the Casting Method [45]

(Composite Samples from 10 or More Sheep) [34]

	High crimp	Low crimp
Base	20.6	18.8
Middle	19.9	21.6
Tip	22.0	23.8

[4] The statistic P represents the probability of an effect occurring by chance. Thus a difference in diameter as great as, or greater than, that found would result more than 4 times out of 10 in sampling from one population.

[5] A similar approach to the same problem recently has been employed by Roberts [35].

TABLE IV. Properties of the Raw Wools

Property	Method	High crimp Mean	High crimp C.V., %	Low crimp Mean	Low crimp C.V., %
Staple crimp, in.$^{-1}$		14.8 ± 1.1	16.1	8.9 ± 0.5	12.2
Staple length, in.	A.S.T.M. D1234–54	3.45 ± 0.08	12.2	3.90 ± 0.06	9.3
Scouring yield, %	A.S.T.M. D1060–55T; 584–54T	61.3		68.5	
Fiber length, in.	[42]	4.35 ± 0.14	23.0	5.33 ± 0.14	18.4
Contour ratios	Sections	1.21 ± 0.02	12.4	1.21 ± 0.02	11.4
Uncrimping ratio	See text	1.34 ± 0.04	13.5	1.37 ± 0.05	16.6

TABLE V. Fiber Friction against Glass [29]

Wool	μ_{TR}	μ_{RT}	$\frac{\mu_{TR}-\mu_{RT}}{\mu_{RT}}$
High crimp	0.353	0.287	0.230
Low crimp	0.378	0.304	0.243
P	0.1		→1

rived on a wide range of Australian wools, are used, a similar result is obtained. Interpolating between Lang's values, 22μ diameter is found to correspond to an average crimp frequency of 8.9. It is evident that the low-crimp wool of this study lies close to the average crimp-fineness relationship, whereas the high-crimp is exceptional in its crimp configuration.

Fiber Friction

Friction against glass was measured by means of the capstan method described elsewhere [29]. Twenty fibers of each wool were tested at 65% R.H., 70° F., using a downward force of 1 g. and a rubbing speed of 0.5 in./min. The coefficients of friction derived from the mean force developed on the fifth rub cycle and the resultant differential friction ($\mu_{TR} - \mu_{RT}/\mu_{RT}$), are reported in Table V. The coefficients of friction against glass may be slightly higher for the low-crimp wool; there is no evidence for any difference between the wools in the differential friction.

Mechanical Properties and Chemical Reactivity

The average tensile stress-strain curves of fibers from the two wools are depicted in Figures 3 and 4. The former represents the behavior in the uncrimping region of the curve, and the latter the complete curve; in each case both the dry (65% R.H.) and wet (pH 9.2) performance is illustrated. The results were obtained on root portions of fibers from the greasy wool, extracted with methylene chloride, alcohol, and water. The values of the principal parameters of the diagram are listed under "zero time in acid" in Table VI, together with the P levels of the between-wool differences.

There is a large difference between the two wools in the uncrimping portion of the curve at 65% R.H.; this difference practically vanishes in the wet state. Beyond the Hookean region, the low-crimp wool appears a little stronger dry; the two curves are completely indistinguishable above the Hookean region in the wet state, except for the possibility of sligthly higher breaking parameters of the low-crimp wool.

In addition to the single-cycle tests, some repeated cycling experiments were performed in the following manner: each fiber was extended to a predetermined strain at a 50%/min. rate of extension and immediately retracted at the same rate.[6] After 1 min., the fiber was re-extended to the same strain. The three properties computed from the resultant diagrams—first cycle hysteresis, set, and second cycle work loss—are indicated in Figure 5.

Such tests were made at 65% R.H. at cycling extensions of 5 and 15%, and in 0.1 M sodium borate at extensions of 10 and 30%; the results are reported in Table VII. In the low-strain (5%) test in the dry state, the low-crimp wool has substantially inferior recovery performance by all three indices—hysteresis, work loss, and set. No difference between the wools is evident in the 15% dry test, or in the wet condition at either of the strains employed.

The data reported so far characterize the tensile properties of the two wools in their intact state.

[6] In order to establish the correct reversal position of the Instron crosshead, it was necessary to determine the "uncrimped length" position first, by extending the fiber into the Hookean region.

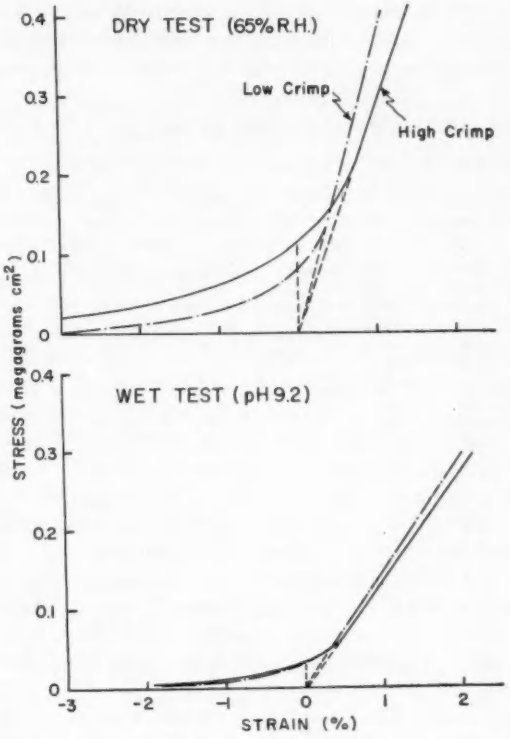

Fig. 3. The low-strain region of the stress-strain curve of intact fibers.

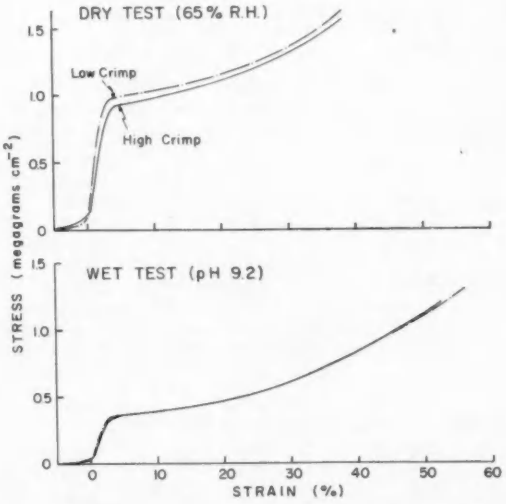

Fig. 4. The full stress-strain curve of intact fibers.

Only minor between-wool differences are observed, except for effects ascribable to the crimp form. It is, however, necessary also to investigate the mechanical properties of the fibers after exposure to chemical treatment, since studies in this laboratory have shown that keratin fibers exhibit a considerable range in the magnitude of their response to chemical attack, such as boiling in acid, as measured by changes in alkali solubility [15], and the fiber tensile properties [14]. These effects have been ascribed to differences in the structure of the fiber cortex [15, 16]. The wools of this study are both crimped, and therefore possess a bilateral cortex [24, 30], but since they differ in their crimp configurations, there must be a difference either in the geometric arrangement of the two cortex components, or in relative amounts present, or in their relative response to physico-chemical environment. The similarity of the two intact wools in their mechanical properties is thus no guarantee of a similar state of affairs in the finished fabric.

A qualitative indication of the stability of the cortex is the temperature of fiber supercontraction in 50% aqueous phenol [48]. The experiments to establish this were made, for simplicity, on yarn. Twenty-centimeter lengths of steamed 1/38's metric yarn (soap-soda scoured in the raw state) marked with a 10-cm. reference portion by means of knots, were immersed in the phenol solution for 20 min., rinsed in water, and dried in the absence of tension. The results, shown graphically in Figure 6, indicate that the low-crimp wool begins to supercontract at a somewhat lower temperature, and thus suggest that the two wools differ in the stability of their cortices.

In order to characterize the properties of the cortex components more closely, the rate of attack

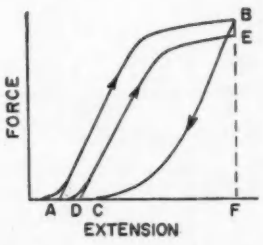

Fig. 5. Diagram of cycling force-extension experiments. % Hysteresis $= \frac{100 \text{ Area ABCA}}{\text{Area ABFA}}$; % work loss $= \frac{100 \text{ Area DEFD}}{\text{Area ABFA}}$; % set $= \frac{100 \text{ AD}}{\text{fiber length}}$

by acid, as indicated by alkali solubility, was determined [14, 17]. In Figure 7, the logarithm of alkali *insolubility* (the complement of alkali solubility) is plotted against the time in boiling 0.04 *N* sulfuric acid. Assuming that two components are present, both reacting with first-order kinetics, their

TABLE VI. Tensile Properties of Intact and Acid-Treated Fibers (15 Fibers per Cell)

Property	Time in acid (hr.)	Dry test (65% R.H.) High crimp	Dry test (65% R.H.) Low crimp	Dry test (65% R.H.) P*	Wet test (pH 9.2) High crimp	Wet test (pH 9.2) Low crimp	Wet test (pH 9.2) P*
Uncrimping stress, megagrams cm.$^{-2}$	—	0.117	0.077	→0	0.032	0.031	—
	4	0.095	0.078	0.05	0.016	0.012	0.001
	16	0.077	0.060	0.05	0.006	0.006	—
Young's modulus, megagrams cm.$^{-2}$	—	28.6	37.2	→0	13.6	14.5	0.2
	4	30.0	34.1	0.1	8.4	6.7	0.01
	16	28.9	33.0	0.1	4.0	3.6	—
Stress at 5% extension, megagrams cm.$^{-2}$	—	0.923	0.991	0.1	0.357	0.357	—
	4	0.878	0.871	—	0.208	0.158	0.05
	16	—	—	—	0.098	0.091	—
Stress at 20% extension, megagrams cm.$^{-2}$	—	1.109	1.164	0.1	0.462	0.460	—
	4	0.994	0.975	—	0.279	0.224	0.05
	16	—	—	—	0.134	0.137	—
Stress at break, megagrams cm.$^{-2}$	—	1.56	1.64	—	1.20	1.30	0.1
	4	1.18	1.07	—	0.59	0.43	0.01
	16	0.61	0.63	—	0.18	0.15	—
Extensions to break, %	—	38.0	38.4	—	52.3	55.9	0.2
	4	30.8	30.1	—	44.7	41.5	—
	16	3.6	2.6	—	25.3	22.2	0.2
Energy to break per unit vol., mg. cm.$^{-2}$	—	0.426	0.463	—	0.341	0.367	0.2
	4	0.301	0.288	—	0.143	0.102	0.05
	16	0.019	0.012	—	0.034	0.027	—

* A dash indicates $P > 0.2$.

TABLE VII. Tensile Cycling Performance of Intact and Acid-Treated Fibers (15 Fibers per Cell)

Property	Time in acid (hr.)	Dry test (65% R.H.) High crimp	Dry test (65% R.H.) Low crimp	Dry test (65% R.H.) P*	Wet test (pH 9.2) High crimp	Wet test (pH 9.2) Low crimp	Wet test (pH 9.2) P*
		5% strain			*10% strain*		
Hysteresis, %	—	51.5	60.8	→0	46.7	46.7	—
	4	60.0	65.4	0.05	43.0	39.0	0.01
	16	—	—		36.4	33.8	0.05
Work loss, %	—	27.5	37.2	0.001	9.3	7.7	—
	4	38.1	47.4	0.001	9.2	8.1	—
	16	—	—		9.9	9.0	—
Set, %	—	0.8	1.2	0.01	0.1	0.1	—
	4	1.2	1.6	0.01	0.1	0.1	—
	16	—	—		0.1	0.1	—
		15% strain			*30% strain*		
Hysteresis, %	—	79.8	80.6	0.05	56.6	55.2	0.05
Work loss, %	—	51.0	54.1	→0	16.8	15.8	—
Set, %	—	5.6	6.3	0.05	6.9	6.9	—

* A dash indicates $P > 0.2$.

proportions and rate constants may be calculated by fitting the equation

$$f_t = F_o e^{-k_o t} + F_p e^{-k_p t}$$

where f_t is the fraction of wool found insoluble after t hours of exposure to acid, proportionality constants F_o and F_p refer to the two components, ortho and para, k_o and k_p being the corresponding rate constants [17].

The constants were determined by a novel method for least square estimation on a nonlinear model.[7] The values obtained and their 95% confidence limits are given in Table VIII; the resultant curves are shown in Figure 7, the dotted lines indicating the intercept and slope of the second (para) portion.[8]

The results for the two wools are better characterized by the two curves than by a mean curve derived from both sets of values (P for residuals about the regression line $\rightarrow 0$). Consequently, they prove that the low-crimp wool has the greater overall sensitivity to acid attack, as measured by the alkali-solubility test. However, the data are insufficient to yield statistically significant differences between the two wools in any of the four constants of the equation; therefore, they do not reveal whether the cause of the difference lies in the relative proportions or in the relative reactivity of the cortex components of the two wools.

The results of single-cycle tensile tests on fibers exposed to boiling 0.04 N sulfuric acid for 0, 4, and 16 hr. are reported in Table VI. The wet test (0.1 M borax) data parallel the alkali-solubility observations, indicating that the low-crimp wool suffered the greater amount of damage for the same extent of acid treatment. No such differentiation between the two wools is evident in the dry test data, which exhibit much smaller changes in the parameters after contact with the acid, except for the catastrophic fall in breaking extension at 16 hr. acid treatment.

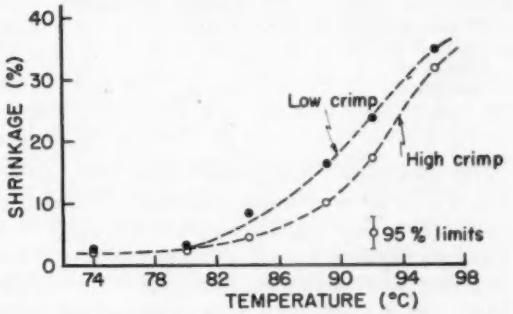

Fig. 6. The supercontraction of yarn in 50% phenol as a function of temperature.

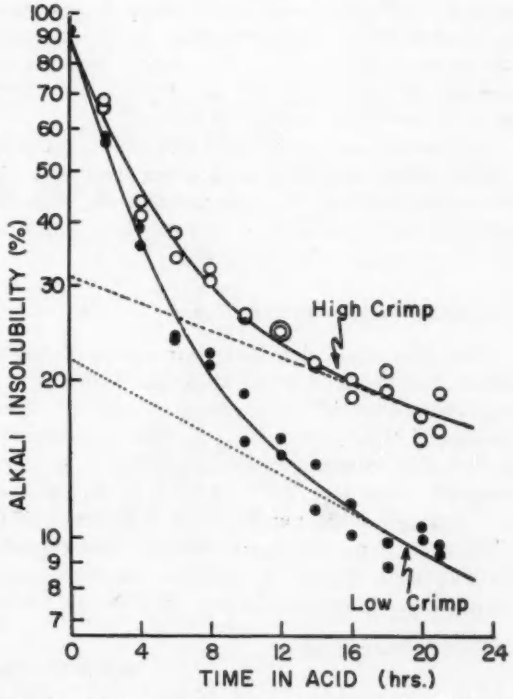

Fig. 7. Alkali insolubility as a function of time of immersion in boiling 0.04 N sulfuric acid.

[7] The method, due to Box (unpublished), is a development of a technique already described (Box, G. E. P., and Coutie, G. E., *Trans. Inst. E.E.* **103B**, Suppl. 1, 100 (1956)). We are indebted to G. E. P. Box for his advice, and to R. E. Heitman for the machine computations.

[8] The values of the constants and their confidence limits, which were derived by a less precise method, are given in a preliminary note on the subject (Menkart, J., and Detenbeck, J. C., *Nature* **178**, 264 (1956)); they differ somewhat from those of Table VIII.

TABLE VIII. Proportionality and Rate Constants for Weight Loss in Alkali after Treatment with Acid

	F_o	k_o	F_p	k_o/k_p
High crimp	0.62 ± 0.20	0.30 ± 0.13	0.31 ± 0.20	0.029 ± 0.037
Low crimp	0.71 ± 0.08	0.33 ± 0.05	0.22 ± 0.08	0.044 ± 0.022

TABLE IX. Cystine Content and Moisture Regain

	High crimp	Low crimp	High/low
Cystine content, %	12.1 ± 0.42	11.3 ± 0.30	1.07
Moisture regain (%) at 65% R.H., 70° F.			
Adsorption	15.3	15.1	1.01
Desorption	16.8	16.6	1.01

The results of cycling tensile tests (10% strain wet, 5% strain dry) parallel the above observations, in the sense that the wet measurements indicate a difference between the two wools in response to acid treatment, and the dry measurements do not (Table VII). In the wet tests, both wools exhibit a decrease in the first cycle hysteresis on exposure to acid, that of the low-crimp wool being significantly greater. (The hysteresis is, of course, not an index of absolute recovery performance.) In the dry state, the recovery behavior of both wools is impaired by the acid to a similar extent, and the original difference between them remains unaltered.

It is shown later in the paper that commercial acid dyeing reveals the difference between the two wools in sensitivity to acid damage, and that the degradation in fiber mechanical properties thus caused is evident in certain fabric properties.

Chemical Composition and Regain

The two wools differ in their cystine content, determined by the Shinohara procedure, that of the high-crimp material being greater (Table IX); the ranking is to be expected on the basis of reactivity toward acid, by analogy with observations of a wider range of animal fibers [15]. Almost complete amino acid analyses of the two wools by Simmonds [37], using the ion exchange–ninhydrin colorimetric method, have shown, in addition, the high-crimp wool to have significantly less glycine and more proline. The high-crimp wool appears to be slightly more hygroscopic.

Processing and Product Evaluation

The two wools were scoured with soap and soda and made into French top (195000 grex), identical processing conditions being employed. Both carded satisfactorily, and yielded similar amounts of noil in combing. The length distributions of the raw wools and tops are illustrated by the histograms of Figure 8, obtained on the single-fiber tester [42]. Considerably more fiber breakage must have occurred in the carding and combing of the low-crimp wool, since its mean fiber length in top is close to that of the high-crimp, in spite of its original length advantage in the staple. In Table X, the single-fiber data are compared with analyses by the comb-sorter method. In other tests good agreement has been observed between the length-biased mean derived from the single-fiber tests and weight-based mean from the comb-sorter [42]; in the present instance the values for the low-crimp wool given by the two methods are in good agreement, but in the high-crimp the comb-sorter mean is appreciably lower than the single-fiber mean. This discrepancy is probably due to incomplete crimp removal in the comb sorter.

The difference in fiber crimp, while still present in the top (see below), has clearly very little influence on the trade quality evaluation. The two tops were classed by an expert appraiser as follows:

High crimp: 64's super warp, excellent color, a sound top with good hand.

Low crimp: 62's–64's super warp, excellent color, good strength and excellent hand.

The half-grade difference was considered by the appraiser as due to the "coarse edge" present in the low-crimp wool, rather than to the difference in fiber crimp [40].

TABLE X. Fiber Length in the Top

Method	Basis	High crimp Mean	High crimp C.V., %	Low crimp Mean	Low crimp C.V., %	Laboratory
Single-fiber tester [42]	Number	2.83 ± 0.13	45.2	2.79 ± 0.15	52.0	A
	Length	3.38 ± 0.11	33.8	3.58 ± 0.15	40.6	
Comb-sorter [A.S.T.M. D595–55T]	Weight	3.04	35.7	3.51	36.1	B
	Weight	3.14	35.5	3.62	36.6	C

The tops were processed into roving of 2700 grex on the French system, and the roving was ring-spun. The mean fiber lengths in the grease, top, roving, and yarn stages are shown in the bar graphs of Figure 9. Clearly, some fiber breakage occurs in drawing, and a little more in spinning; the extent of breakage must be greater than the changes in mean fiber length seem to indicate, as the material lost between the stages in the form of fly and waste consists largely of short fibers.

The force-extension properties of the top and roving were examined on the Instron tester [7, 29]. The mean curves are shown in Figure 10. Those for top were obtained by testing 20 strands of each wool type after 60 days aging in the package; the roving curves are derived from the data for 20 strands of each type (representing 5 bobbins), tested one day after processing.

The two wools are differentiated in the same manner in the two assemblies, but the effect is much more striking in the case of roving. It has been postulated that the first, rising portion of the curve is due principally to crimp deformation; when fiber slippage begins to predominate, the resistance to further extension decreases [7]. The present data, showing that with greater crimp content a much higher maximum load is reached, at a somewhat higher extension, supports this mechanism. In fact, the ratio of the maximum tenacities of the rovings is only a little smaller than the ratio of the fiber uncrimping stress (Table VI); this suggests that essentially all the force developed in the first stages of roving extension is due to fiber decrimping, rather than to fiber re-alignment. The roving tenacity is an order of magnitude lower than the uncrimping tenacity of its constituent fibers, which indicates that only a small fraction of the fibers present is participating in the process (or that each fiber contributes only a small fraction of its uncrimping energy). In the top stage, the tenacity is a little greater, and the difference between the two wools is smaller than in roving. This is probably due to poorer fiber parallelization in the top, resulting in modes of fiber deformation other than decrimping playing a part in the process.

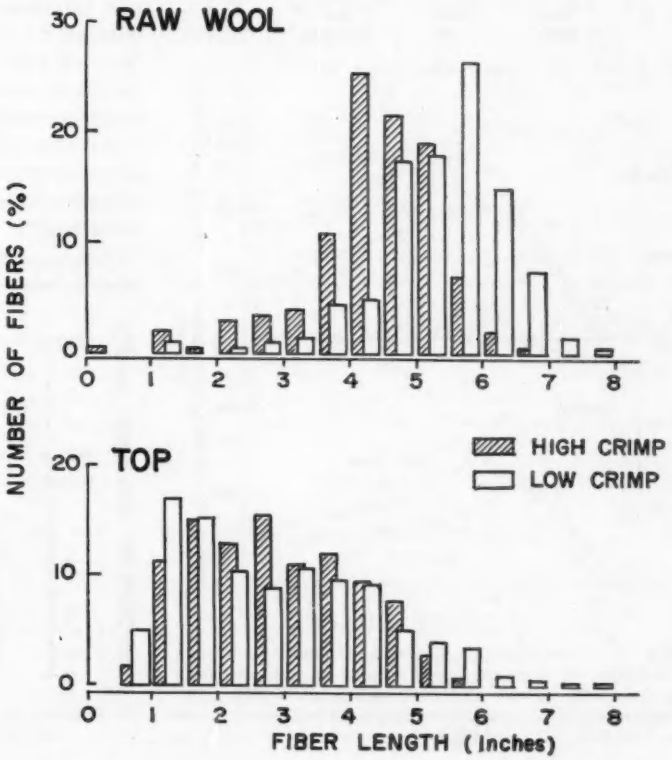

Fig. 8. Fiber length distributions of the raw wool and top.

TABLE XI. Counts and Spinning Test Conditions

Metric counts	Grex	Twist (cm.$^{-1}$)	Twist constant*	Amount spun (kg.)	Spindle-hours
1/45's	222	6.5	0.93	21	1700
1/38's	263	8.0	1.68	40	2600

* $\frac{(\text{turns/cm.})^2}{\text{metric counts}} = \frac{(\text{turns/cm.})^2\,(\text{grex})}{10^4}$. A value of 0.5 represents a very soft yarn, 1.5 an average warp twist, and 3 a very hard yarn.

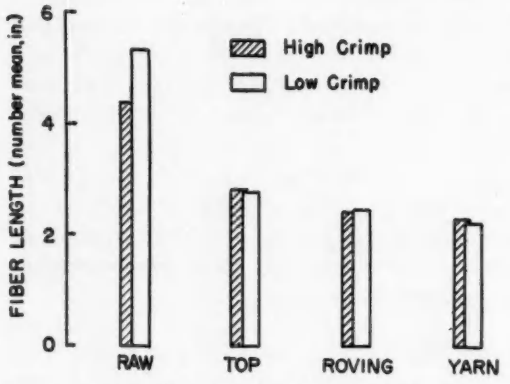

Fig. 9. Changes in mean fiber length through processing.

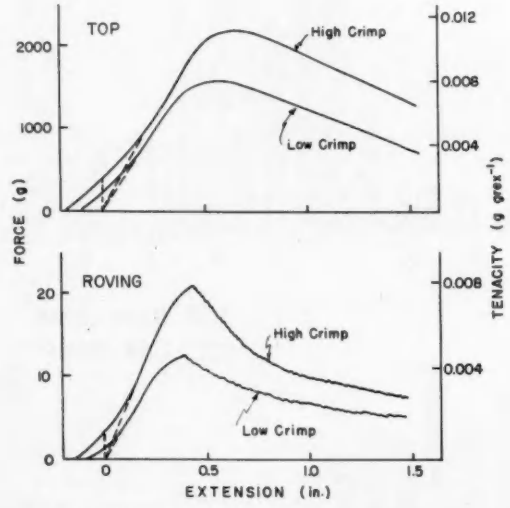

Fig. 10. Force-extension curves of twistless strands: (a) French top, 195 k/grex, gauge length 10 in., rate of extension 5 in./min. (P for the main parameters = 0.05); (b) French roving 2.7 k/grex, gauge length 15 in., rate of extension 5 in./min. ($P = < 0.001$).

The phenomena just discussed are concerned only with the first few per cent of extension; on further stretching of a twistless strand, attenuation in one narrow zone takes place. As might be expected, crimp has an influence on the cohesion of the fibers in the slippage zone. Under the above testing conditions, complete rupture of the low-crimp rovings took place at an extension of about 3½ in., and of the high-crimp ones at about 4½ in.

Aging of the roving on the bobbin for 49 days produced the following changes in the force-extension properties of the strand: no change in the force at zero extension (as defined by the Hookean slope); an increase in the slope; a slight increase in maximum load; and a decrease in extension to maximum load. These effects, which are in accord with earlier observations [29], ran in parallel on the two wools.

Spinning Performance

Two counts of yarn were spun; the details, together with the quantities involved, are given in Table XI.

In each case one frame-side was used, and split into four blocks among which the two wools were distributed. The spinning of each count occupied two full doffs, and the positions of the rovings on the frame were interchanged on doffing. The end breakage results are shown in bar-graph form in Figure 11. The high-crimp material clearly performs better near the limit of spin; the effect can undoubtedly be ascribed to the greater cohesion of the roving.[9]

[9] In fact, the difference between the wools in spinning performance may be greater than these data indicate. The

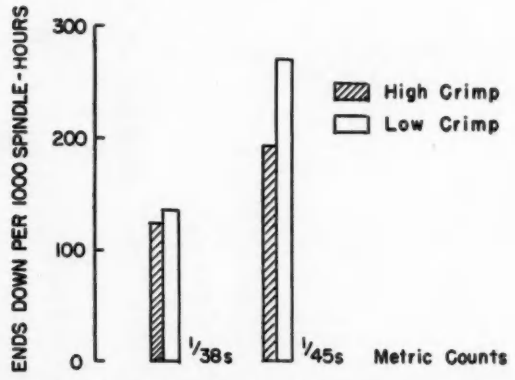

Fig. 11. End breakage in spinning. P for wools < 0.01; wools × counts 0.1 (χ^2 test).

The yarns from the two wools were indistinguishable in their "percentage unevenness," as determined on the Uster instrument. A small difference was observed, however, in the pattern of irregularity as recorded on the Uster Spectrograph (for the 1/45's yarn). The peak on the variation intensity–wave length curve occurred at a wave length of about 8 in. in the high-crimp yarn, and 10–12 in. in the low-crimp yarn (P for the difference between the curves $= 0.1$). It has been suggested that the shape of the curve is a function of the fiber length, with the peak occurring at a constant multiple of the mean length [20]. The two wools have the same mean fiber length, but as already noted, the fiber *extent* in the yarn may well be smaller for the high-crimp wool, and this could be the operative factor. It seems unlikely, however, that the difference between the irregularity patterns is sufficient to affect the appearance of the yarns, or of the fabrics made from them.

Yarn Properties

Small quantities of 1/38's metric yarn were prepared at different twist levels for the examination of the bulk density and the tensile properties of the yarns. The bulk density was determined by measuring the thickness of a parallel array of 200 yarns (steamed, and soap scoured in the skein) placed within a square-based slot with a $\frac{1}{2}$-in. square base. A rectangular brass block, fitting loosely within the slot, was placed on top of the material, and the thickness was measured after 1 min.; another weight was added and the thickness was remeasured after another minute. The yarns were then cut off flush with the sides of the slot by means of a razor blade and weighed. The bulk density under the two pressures employed is shown as a function of the twist constant in Figure 12.[10] The high-crimp yarn has the lower bulk density at normal twist levels, but the difference vanishes at very high twist; the curve for the higher pressure is very similar to that for the lower, which suggests that the lower bulk density of

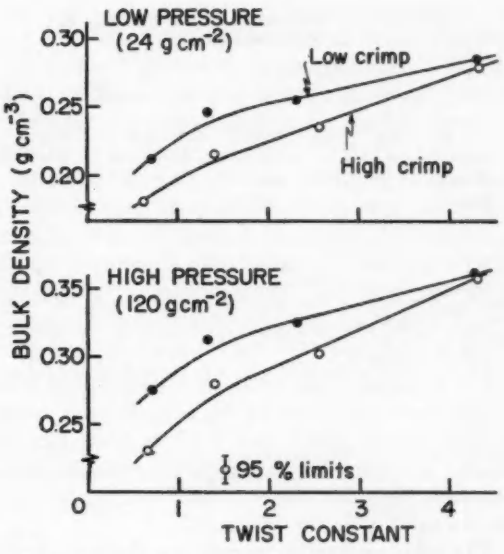

Fig. 12. The bulk density of 1/38's metric yarn as a function of twist.

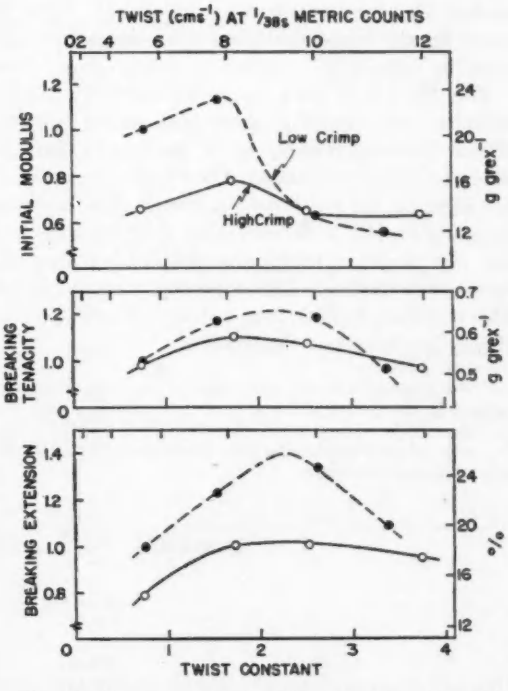

Fig. 13. Yarn modulus, breaking tenacity and breaking extension as a function of twist. (On the left-hand ordinate, low-crimp at 0.75 twist constant = 1.)

average number of fibers per yarn cross section depends on the effective linear density of the fibers. The latter is related not to the straight fiber length, but to the fiber extent along the yarn axis. If the two wools differ in this respect, the extent will undoubtedly be smaller in the high-crimp fibers, consequently the high-crimp yarn will contain fewer fibers per cross section than a low-crimp yarn of the same linear density.

[10] Cf. footnote to Table XI. The constant is used, rather than the twist in turns per unit length, to correct for small count variations among the samples.

TABLE XII. Probability Levels of Effects in Yarn Properties

Property	*P*		
	Wools	Twists	Wools × twists
Modulus	→0	→0	<0.001
Tenacity	0.1	0.01	→1
Breaking extension	0.01	0.1	→1

the high crimp is not due to the presence of more surface hairs, but to a basic difference in the geometry of the strand.

The force-extension behavior of the steamed yarn (in oil) was determined on the Instron tester, using a 10-in. gauge length and a 12 in./min. rate of strain. Twelve strands originating from six bobbins were tested for each wool and twist; each strand was weighed after breakage.

The initial part of the curves was linear from the origin, no "uncrimping region" being evident. The results for three properties—initial modulus, breaking tenacity,[11] and breaking extension—are plotted against the twist constant in Figure 13. The *P* levels for the main effects and their interactions are listed in Table XII.

The low-crimp yarn has a considerably higher modulus and breaking extension, and a slightly higher breaking tenacity, over the greater part of the twist range examined. The "optimum twist" is not identical for the three parameters, the modulus dropping rapidly with increasing twist constant before the maximum tensile strength and breaking extension are reached. This aspect of yarn properties does not seem to have been noted previously; it obviously has important implications.

[11] The tenacity was derived from the *maximum load* developed in the force-extension test; this coincided with the breaking strain, except at the lowest twist level (0.75, i.e., 13 t.p.i.) where considerable fiber slippage occurred in the later stages of extension.

In Table XIII, the properties of the "optimum yarns" (from the curves of Figure 13) are compared with those of the fibers withdrawn from them (converted from data of Table XIV). The difference between the two wools in yarn breaking tenacity is almost completely accounted for in terms of their fiber tenacity. In modulus, on the other hand, the fiber property/yarn property ratio (transmission coefficient) is much lower for the high-crimp wool; the effect is readily explicable by the lower compactness of this yarn. A striking feature of the modulus data is the very high transmission coefficient of the low-crimp wool. A 97% translation of fiber property into staple yarn property is impossible to credit; it is obviously an artifact, resulting from the difference between the gauge length of the single fiber test and the "effective gauge length" of the fibers in the yarn [44].

The higher breaking extension of the low-crimp yarn suggests that more fiber slippage occurs in the course of its stretching. However, such a process should be reflected in the breaking tenacity also; the discrepancy is at present unexplained.

Changes in Fiber Tensile Properties Through Processing

At different processing stations, fibers were withdrawn from the assemblies and subjected to force-extension tests at 65% R.H. and in borate buffer, pH 9.2; the low-strain tests at 65% R.H. were performed within 24 hr. of withdrawal from the assembly. In order to determine the stability of any changes in the uncrimping portion of the stress-strain curve, some fibers were relaxed prior to testing by immersion in cold water or by exposure to steam at 60° C., 85% R.H. for 10 min. in the absence of constraint. The data for the principal parameters at various processing stages are summarized in Table XIV. The levels of uncrimping stress and elastic modulus in the raw wool, top, yarn,

TABLE XIII. The Properties of Fibers and Optimum Yarns

	High crimp			Low crimp		
	Fibers	Yarn	Transmission coefficient, %	Fibers	Yarn	Transmission coefficient, %
Initial modulus, g. grex^{-1}	22.7	16.0	70	23.8	23.0	97
Breaking tenacity, g. grex^{-1}	1.09	0.59	55	1.13	0.65	58
Breaking extension, %	35.6	18.8	53	36.0	25.4	71

and ready-to-dye and dyed fabric are indicated by the bar graphs of Figure 14.

The uncrimping stress of the high-crimp wool diminishes progressively through drawing and spinning; this decrease is in the form of temporary set, as relaxation of either the yarn or of fibers withdrawn from the yarn results in recovery to almost the original value. The uncrimping stress increases on passing from yarn to the ready-to-dye fabric, and does not change any further in dyeing and finishing. This new level is stable to relaxation by wetting or steaming.

The uncrimping stress of the low-crimp wool remains practically unaltered through topmaking, drawing, and spinning. A radical increase is observed in the fabric, the new value being considerably higher than that in the original wool, and practically identical with that of the high-crimp fibers at the same stage; the value attained is unaffected by wetting or steaming.

The data suggest that the fabric crimp has been impressed onto the fibers and is making a contribution to their mechanical properties comparable to that of the original fiber crimp. Visual examination shows this to be the case. The fibers shown in Figure 15 were withdrawn from the assemblies and subjected to the same small tension.[12] An appreciable reduction in the crimp level between the raw

TABLE XIV. Changes in Fiber Tensile Properties through Processing. (Fifteen or More Fibers per Cell.) All Data from the Fabric Stage are for Fibers Withdrawn from the Warp Yarn

		Dry (65% R.H.)			Wet (pH 9.2)		
Property	Stage	High crimp	Low crimp	P**	High crimp	Low crimp	P**
Uncrimping stress, megagrams cm.$^{-2}$	Raw	0.117	0.077	→0	0.032	0.031	—
	Top	0.096	0.073	0.001			
	Yarn	0.085	0.076	0.01			
	Yarn, relaxed*	0.096	0.078	0.01			
	RTD	0.101	0.097	—	0.029	0.028	—
	RTD, relaxed*	0.099	0.099	—			
	Tan	0.101	0.103	—	0.022	0.019	—
	Tan, relaxed*	0.103	0.104	—			
Young's modulus, megagrams cm.$^{-2}$	Raw	28.6	37.2	→0	13.6	14.5	0.2
	Top	28.9	32.1	0.1			
	Yarn	30.0	31.4	—			
	Yarn, relaxed*	28.3	32.3	0.05			
	RTD	30.9	31.3	—	11.6	11.7	—
	RTD, relaxed*	30.6	31.1	0.2			
	Tan	29.5	28.9	—	7.8	7.5	—
	Tan, relaxed*	29.5	28.7	0.2			—
	Navy	29.1	29.8	—	9.7	9.0	0.2
Breaking stress, megagrams cm.$^{-2}$	Raw	1.56	1.64	—	1.20	1.30	0.1
	Yarn	1.44	1.49	—			
	RTD	1.47	1.57	—	1.20	1.24	—
	Tan	1.17	1.10	—	0.67	0.57	0.2
	Navy	1.18	1.19	—	0.87	0.80	—
Extension to break, %	Raw	38.0	38.4	—	52.3	55.9	0.2
	Yarn	35.6	36.0	—			
	RTD	30.3	32.1	—	48.6	51.3	—
	Tan	22.8	20.9	—	42.8	41.5	—
	Navy	22.0	23.8	—	46.6	45.0	—
Breaking energy per unit volume, megagrams cm.$^{-2}$	Raw	0.426	0.463	—	0.341	0.367	0.2
	Yarn	0.352	0.364	—			
	RTD	0.322	0.360	—	0.293	0.38	0.1
	Tan	0.221	0.199	—	0.157	0.136	0.05
	Navy	0.214	0.232	—	0.212	0.188	0.2

* Relaxed: Fibers exposed to steam at 60° C., 85% R.H., in the absence of tension, for 10 min.
** A dash indicates $P > 0.2$.

wool and the yarn stage is evident, as is the appearance of the new, regular crimp form in the fibers withdrawn from the fabric. The last group ("Tan Relaxed") consists of fibers immersed in water at 60° C. for 10 min. and allowed to dry without tension; the unchanged fiber configuration indicates the stability of the newly imposed fabric crimp. Both the fiber tensile properties and appearance of the fibers suggest, therefore, that the differences in crimp content present in the original wool have been eliminated or masked in manufacture.

The changes in the tensile properties beyond the uncrimping region, shown in Table XII, do not call for much comment. In the wet test, the low-crimp wool has, after dyeing, the lower value in all the measured parameters of the stress-strain curve, thus reversing the position in the earlier stages. The observation is in accord with the already noted greater sensitivity of this wool to attack by acid.

Fabric Properties

The fabric made was a charmeen, a material of the gabardine type, the weave being a 3/2 twill, stepped two up, with 1/38's metric warp and 1/45's filling. The fabric received a clear finish, and the finished particulars were 102 ends, 86 picks/in., weight 0.024 g./cm.^{2} (11 oz./linear yd., 56 in. wide). The qualitative examination of the fabric and the measurements of fiber and fabric properties were performed in the ready-to-dye (blown and scoured) and in the dyed and finished state. Two shades were dyed, a tan and a navy; both were produced with chrome complex dyestuffs of the 1 : 1 type, and in each case fabrics from both wools were dyed together in the same dye bath. (A very slight difference in the shade between the two wools resulted, the low-crimp being darker in both cases). The tan had a longer dyeing time; the consequences are evident in the fiber tensile properties (Table XIV).

Handle and Appearance

Expert examination classified the high-crimp fabric as the softer, "kinder" handling and smoother, "quieter" in appearance, of the two [40]. The difference, while quite definite, was not considered as

[12] A 7-mg. weight was attached to the lower end of each fiber; the uncrimping force of these fibers is 200-400 mg.

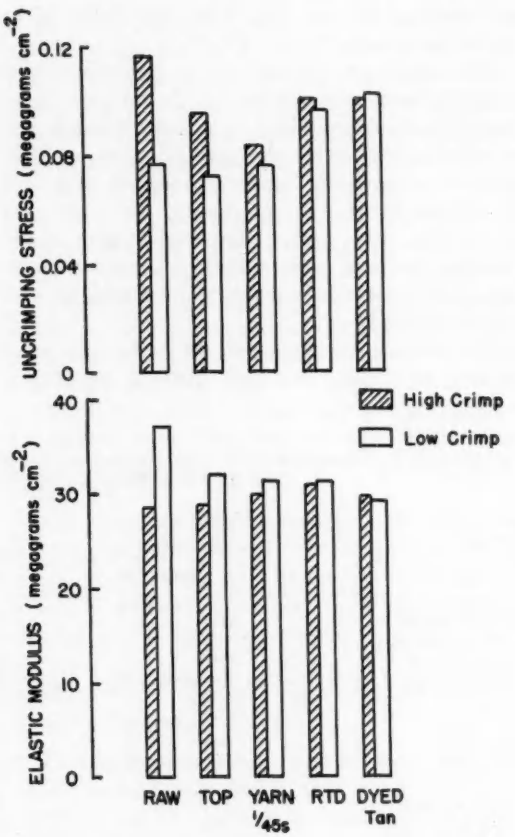

Fig. 14. Changes in fiber uncrimping stress and elastic modulus through processing.

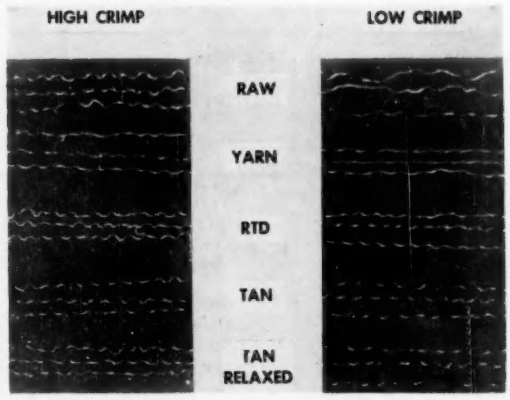

Fig. 15. Fiber crimp configuration at different stages of manufacture (× 1.7).

great as might be expected from two wools assessed in the grease as 64's and 58's–60's, respectively.

The fabrics were also graded for softness by a non-expert panel at the Western Utilization Research Branch of the United States Department of Agriculture; the high-crimp material was judged to be softer [38].

Fabric Thickness

Fabric thickness as a function of pressure was measured on the Instron tester, using the device of deMaCarty and Dusenbury [11]. The fabric was compressed at a rate of 0.02 in./min. with a pressure foot of 1½-in. diameter until the full weight of the piston (340 g., i.e., 29.8 g./cm.²) was applied. The piston was allowed to rest on the fabric for 1 min., after which time the crosshead was retracted at the same rate. One minute later the fabric was recompressed.[13]

Tests were run on the fabric as received, and also on samples relaxed by wetting out in cold water and drying in the absence of tension. (The treatment had negligible effect on fabric area.) Three runs were made on separate portions of each sample. The mean results for thickness at two pressures, measured on the second compression cycle, are recorded in the bar graphs of Figure 16. Comparison with the data of the first cycle showed an appreciable lowering in the thickness at the low load (greater for the wetted out than the control fabrics and similar for both wools), and a very small decrease under the high load.

In all the pairs, the high-crimp fabric is thicker than the low-crimp ($P \rightarrow 0$). The results on the relaxed samples are the more consistent, indicating that the true differences in fabric bulk has to some extent been obscured by the pressing and setting procedures of finishing. Relaxation has restored the thickness of the finished fabrics under low load to near the RTD value.

The difference in fabric thickness between the two wools is clearly due to the difference in yarn bulk, as may be seen from Figure 17, where yarns withdrawn from the finished navy fabric are shown.

Low power micrographs of the fabric itself (Figure 18) do not show such a striking differentiation; nevertheless, it is evident that the high-crimp material has smaller interstices between the yarns, and somewhat more surface cover.

Thus we have a paradoxical situation: the two wools are clearly different in their fiber crimp in the

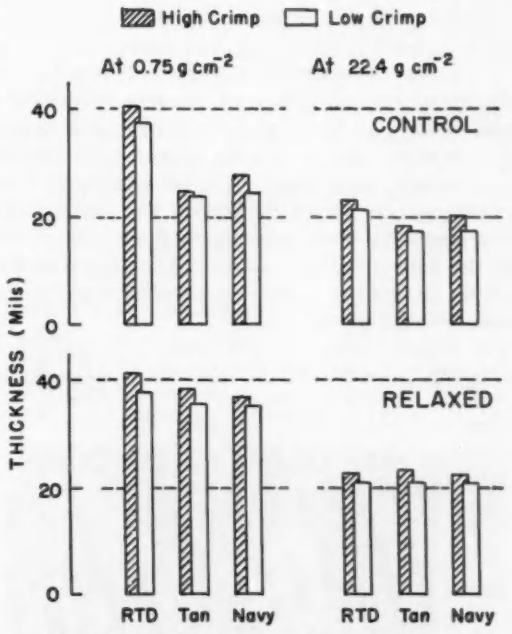

Fig. 16. Fabric thickness under two pressures (second cycle of compression).

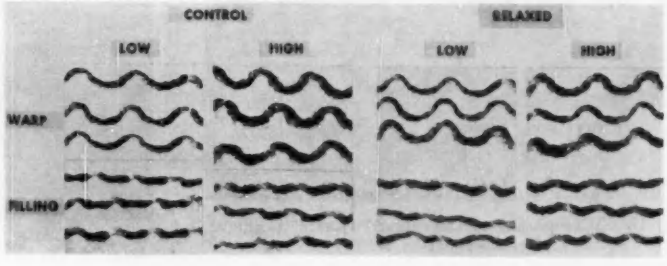

Fig. 17. Yarns withdrawn from navy finished fabric (× 12).

[13] In this work, an Ames gauge reading to 0.001 in. was used in place of the Instron dials to determine the crosshead position.

raw state, whether defined mechanically or geometrically. In the fabric, fibers from the two wools appear identical both in their stress-strain behavior in decrimping and in their crimp configuration, yet differences in the thickness of the fabrics and their constituent yarn are observed, which must be ascribed to a difference in the fiber crimp form. Clearly, some of the original fiber crimp is still present in the fabric stage, though its presence is masked by the superimposed fabric crimp; more refined tools than have been used in this investigation would be required to separate the two components.

Compressional Resilience

Two aspects of energy dissipation in compression were explored:

1. Using the apparatus described above, the change of load with time at constant compression was measured. Because of the variations in thickness between and within the fabrics, each sample was initially compressed to a predetermined load rather than to a fixed thickness. The stress decay was essentially linear with log time over the period of the experiment (12 min.). The slopes of the curves for the two wools did not differ significantly from each other.

2. Repeated cycling experiments were performed using the procedure of the thickness measurements, but reversing the crosshead immediately on reaching the selected maximum load. There was no significant difference between the two wools in either of the parameters measured, first-cycle hysteresis and second-cycle work loss.

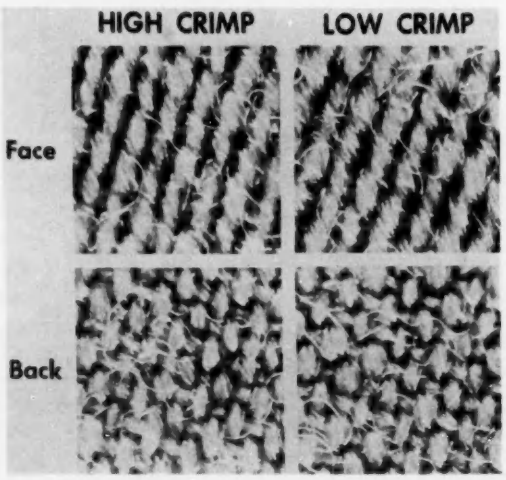

Fig. 18. Micrographs of tan relaxed fabric. (Oblique surface illumination, × 8.6.)

Air Permeability

Measurements were made by the A.S.T.M. method (D737-46) on relaxed samples of the finished fabrics at 65% R.H., 70° F., using a pressure drop of 0.5-in. water. The results, illustrated by Figure 19, indicate that the high-crimp fabric offers less resistance to air flow (P for wools < 0.001). This observation is surprising, as the filling up of the fabric interstices due to the greater yarn bulk (Figure 18) would be expected to have the opposite effect.[14] This factor must be outweighed by the lower over-all bulk density of the high-crimp material.

[14] A separate determination, on different samples, performed in the laboratory of one of the reviewers of the paper, has confirmed these results.

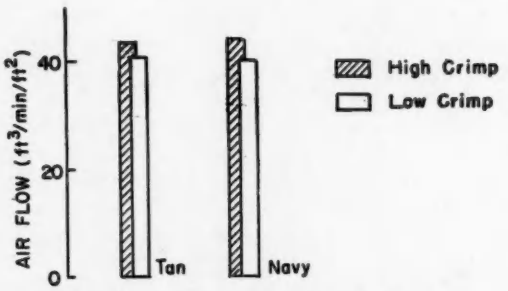

Fig. 19. Air permeability of the finished and relaxed fabrics.

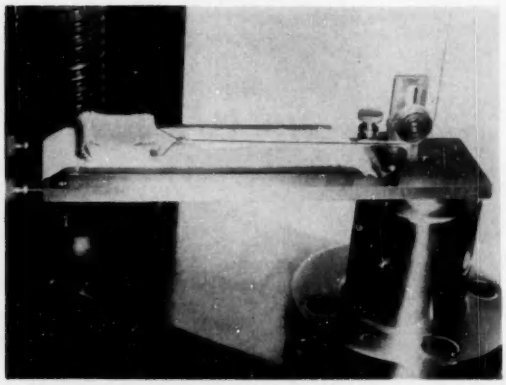

Fig. 20. Device for fabric friction measurement.

Surface Friction

The device used for fabric friction measurement is shown in Figure 20. A 1-in. raveled strip is attached to an aluminum boat with a 1-in. square base; the boat slides over a horizontal surface mounted on the crosshead of the Instron tester. A fine wire connecting the boat, by way of a pulley on the crosshead, with the load cell translates the downward motion of the crosshead into horizontal movement of the slider.

Three test surfaces were used (machined aluminum, vegetable-tanned calfskin, and the fabric itself). All the experiments were made on the fabric face, in the warp direction, at a speed of 5 in./min. Stick-slip motion took place under these conditions; the mean maximum force developed in each run was recorded. Two loads on the slider, 18 and 118 g., were employed. The coefficients of friction (averages for tan and navy relaxed fabrics) are recorded in the bar graphs of Figure 21.

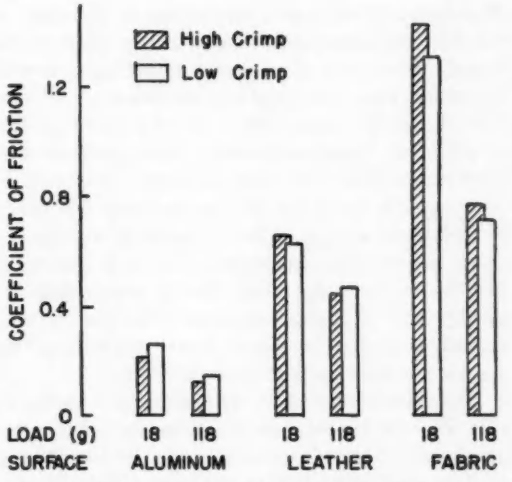

Fig. 21. Fabric friction (mean values for RTD and finished fabrics). *P* for wools: on aluminum 0.05; on leather 0.2 (wools × loads 0.05); on fabric < 0.001.

Against the metal surface, the low-crimp fabric has the greater friction; this is probably a reflection of the fiber frictional properties. When the fabric is rubbed against itself, the high-crimp material exhibits slightly, but significantly, more friction. This is undoubtedly due to the greater extent of fiber interlocking in the more open structure. The leather surface was included in the test as the best available approximation to human skin. In rubbing against leather, the relative rating of the two wools is reversed at the two loads used. The results therefore give no guidance concerning the part played by friction in handle evaluation, since the pressure exerted by the fingers in examining a fabric surface undoubtedly varies and the range of the variation is not known.

Flexural Rigidity and Drape

Relaxation of the fabrics by wetting out produced a curling tendency at the edges, which led to difficulties in bending length and drape measurement and to poor reproducibility in the results. The data quoted below were obtained on unrelaxed material.

Bending length was determined on 1 in. wide strips by means of Pierce's cantilever test. The results for the ready-to-dye and the two dyed fabrics

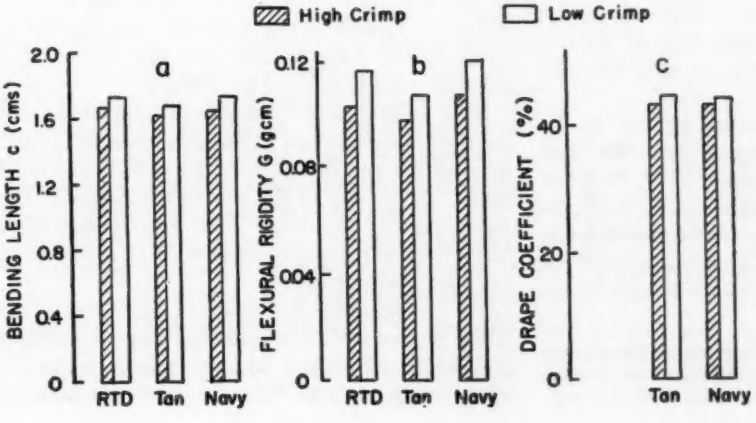

Fig. 22. The bending length and flexural rigidity of the fabrics, determined by the cantilever test, and the drape coefficient, measured on the Drapemeter. *P* for wools: bending length < 0.001, drape coefficient 0.02.

TABLE XV. Properties of 1/38's Me Yarn and of RTD Fabric in the Warp Direction

	High crimp		Low crimp	
	Yarn	Fabric	Yarn	Fabric
Linear density per strand, grex	288	326	280	314
Initial modulus, g. grex^{-1}	16.0	4.2	23.2	4.9
Breaking tenacity, g. grex^{-1}	0.59	0.74	0.62	0.83
Breaking extension, %	18.6	64.4	22.6	66.1

(averaged for both fabric faces and yarn directions) are shown in Figure 22a; the bending length of the low-crimp fabrics is some 4% greater than that of their high-crimp counterparts. The flexural rigidity of the fabrics ($G = c^3w$, where c is the bending length and w the weight per unit area) thus shows a difference of about 12% between the two wools (Figure 22b). The magnitude of the difference is slightly greater than the upper limit of the possible contribution of fiber diameter, which is 10.7% (cf. Appendix B).

It therefore appears that the flexural rigidity of fabric is lowered by the presence of fiber crimp. The data on which this conclusion is based are of course subject to experimental error; but bearing in mind that the diameter contribution as evaluated is likely to be an overestimate of the real effect, the statement is made with some confidence.

The drape coefficients of the finished fabrics, determined on the Drapemeter [10], are indicated in Figure 22c. The high-crimp wool has the lower coefficient (which denotes superior drapability), the difference between the two wools being *ca.* 2½%, i.e., somewhat smaller than that in bending length.[15] The result is important with respect to the appearance ranking of the two fabrics; a 5% difference in drape coefficient represents the minimum class interval detectable by subjective observation [22]; hence, the 2½% difference in drape between the two fabrics is insufficient to influence the subjective visual evaluation.

Thus the difference in flexural rigidity of the fabrics, whether it stems primarily from the fiber diameter effect or from the crimp effect, does not contribute to the fabric *appearance* rating. The possibility that the flexural rigidity difference influences the *handle* rating cannot be so readily dismissed; in the authors' opinion, the relative bulk density of the fabric is the overriding factor.

[15] Fabric bending stiffness will have a smaller effect on the drape rating of a circular specimen than on the bending length of a rectangular strip [22].

Tensile Properties

Force-extension tests were made on 1-in. raveled strips of unrelaxed fabric at a gauge length of 4 in., and a 2 in./min. rate of extension. The "wet" tests were performed on samples soaked overnight in 0.1 *M* sodium borate, stretched immediately after removing from the solution and blotting off the excess liquid. Four tests in each yarn direction were made for every fabric stage and test condition.

In Table XV, some indication is given of the level of the fabric tensile properties relative to that of the yarn from which the fabric is made. The data for yarn are derived from the single-strand test on the 1/38's Me counts, 8 t.p.cm., performed on material from the spinning package; the fabric values are for the ready-to-dye state, in the warp direction, at 65% R.H., 70° F. The linear density of the warp threads under test was used as the normalizing factor for the modulus and tenacity.

The yarn in the fabric is in a crimped configuration imposed by the weave interlacing. The extension of the fabric therefore involves yarn bending as well as stretching; this factor undoubtedly accounts for the fall in modulus from yarn to fabric, and largely also for the increase in breaking extension. The increase in breaking tenacity may be ascribed to the improved cooperation between the fibers in the yarn, caused by the constraint imposed on the yarns by the filling threads; the effect can be considered as a decrease in gauge length.

This constraint is undoubtedly also responsible for the large reduction in the difference in modulus between the two wools from the yarn to the fabric stage. (The ratio of the modulus values is 1.45 in yarn, and 1.17 in fabric.) The greater bulk of the high-crimp yarn cannot exert its full effect in the compact fabric structure.

The relationship of the tensile properties of the fabric to those of its constituent fibers is indicated graphically in Figure 23; the relative values of fabric initial stiffness, breaking load, and breaking

TABLE XVI. Strains Employed in Cycling Force-Extension Tests

	Dry test (65% R.H.)	Wet test (pH 9.2)
Fibers	15	30
Fabric Warp	30	40
Filling	15	27

energy [16] are plotted against the corresponding properties of fibers withdrawn from the fabric (modulus, breaking stress, and breaking energy).

The initial stiffness of the high-crimp fabric at 65% R.H. is in all three states (RTD, tan, and navy) lower than that of its low-crimp counterpart, although their fiber moduli are practically identical. However, as noted above, the between-wools difference in the fabric property is not as large as that observed in yarn. For both wools, the fabric stiffness drops appreciably from the ready-to-dye to the finished state, although the fiber modulus is unaltered. The anomaly is probably due to changes in the geometry of the fabric caused by finishing; its absence in the wet test suggests that these changes are in the form of temporary set.

For initial stiffness tested wet, and for the other two properties under both testing conditions, a linear relationship appears to exist between the fabric properties and the fiber properties, with the values for both wools falling on one line. Thus, it may be concluded that the level of these properties is unaffected by the fiber crimp configuration.

Cycling Performance in Extension

The cycling experiments were performed using the sample size and general conditions of the tensile tests. Each sample was extended to approximately half its breaking strain, immediately retracted, and re-extended 5 min. later. Similar tests were performed on fibers withdrawn from the fabric. The cycling strains employed are listed in Table XVI. The fabric set and work loss are plotted against the corresponding fiber properties in Figure 24.

In the dry tests, anomalous behavior analogous to that observed for the modulus is evident, more strikingly in the case of set: in passing from ready-to-dye stage to the Navy finished fabric, the fabric

[16] The fabric data represent the averages for both yarn directions.

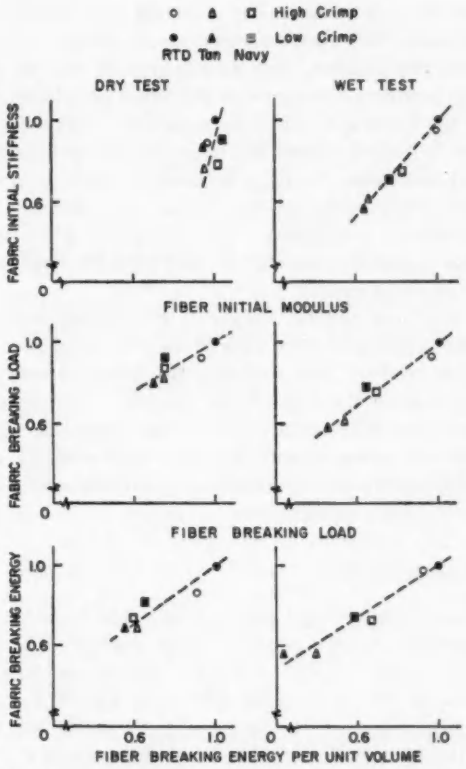

Fig. 23. Fabric tensile properties plotted against the corresponding fiber properties. (Low-crimp ready-to-dye = 1.)

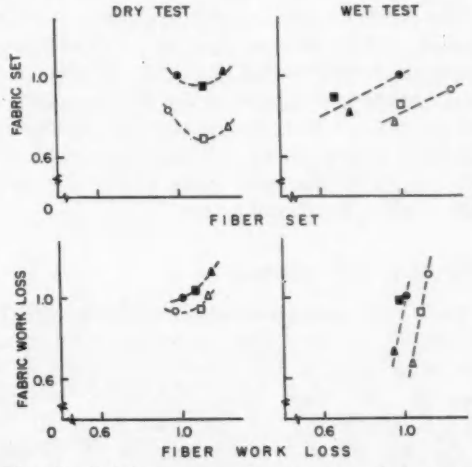

Fig. 24. Fabric second cycle set and work loss plotted against the corresponding fiber properties. The symbols have the same meaning as in Figure 23. (Low-crimp ready-to-dye = 1.)

recovery from extension improves (set decreases), although the fiber recovery is impaired. Further fiber modification, such as occurred in the tan dyeing, produces a response in the fabric paralleling that of the fibers. No such discrepancy is evident in the wet test data, where the improvement in fiber performance after dyeing is reflected in fabric behavior. The relationship appears linear (the data are insufficient to pronounce on this point with confidence), but the slope of the line differs substantially for the two indices.

The most notable feature of the cycling performance is the clear separation of the two wools: for the same level of fiber recovery, the fabric recovery is superior in the high-crimp material. Presumably, the lower bulk density of the high-crimp fabric results in fewer or smaller inter-fiber contacts and consequently in less frictional hindrance to the recovery from deformation.

Wrinkle Recovery

Three samples per fabric were tested in each yarn direction by the vertical strip method (ASTM D1295-53T, AATCC 66-56). Measurements were made at two relative humidities, 65 and 80%, with results indicated in Figure 25. A small, but statistically significant, difference between the two wools was observed at 65% R.H., the high-crimp wool being superior; at the higher humidity, the two wools were indistinguishable.

The difference between the two fabrics in wrinkle recovery at 65% R.H. is about 3½%; the extensional experiments had resulted in a 10% difference in the strain recovery computed in the same manner, and the compression tests had detected no difference at all. Such a divergence is not disturbing, as the fiber deformation in the three types of test differs not only in form, but also in extent.

Flex Abrasion Resistance

The Stoll-Quartermaster instrument (ASTM 1175-55T) was used, the number of cycles to rupture being taken as the index of resistance to flex abrasion. For each fabric and testing condition, three samples were run in each yarn direction.

The capacity of a textile material to withstand abrasion depends on the energy-absorbing ability of its fibers, *ceteris paribus* [23]; it has, in fact, been shown, for one worsted fabric subjected to a series

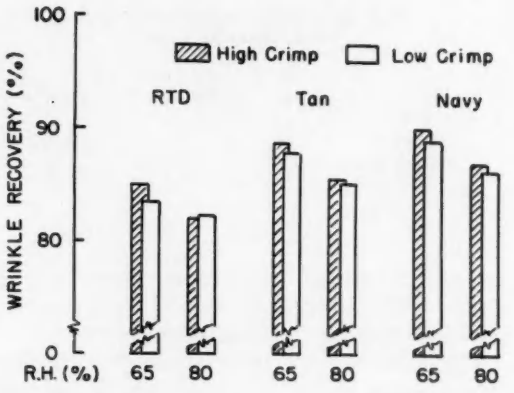

Fig. 25. Wrinkle recovery at 65% and 80% R.H., vertical strip method. *P* for wools at 65% R.H., 0.05; at 80% R.H., > 0.2.

of chemical treatments [17], that resistance to flex abrasion is closely related to the single-fiber breaking energy.[17]

The wet flex abrasion resistance is plotted against the wet fiber breaking energy in Figure 26a. The relationship is clearly linear, and the fact that the points for both wools fall in one curve indicates that fiber crimp has no effect on the performance of the fabric in the flex-abrasion test.

A similar plot for data obtained at 65% R.H. yields a completely different result (Figure 26b); the resistance to abrasion of the finished fabrics is as great as, or greater than, that of the ready-to-dye material, and no relationship to the fiber properties is apparent. (The flex abrasion data also have a high replication variance.)

It was suspected that the high abrasion resistance of the dyed fabrics was due to changes in the fiber surface, possibly resulting from the absorption of surface-active agents from the dye bath. This hypothesis appears valid, because after solvent extraction (methylene chloride, ethyl alcohol, and water), the abrasion resistance of the dyed fabrics drops markedly, while that of the ready-to-dye material remains practically unaltered, and the new values show the expected relation to fiber breaking energy (Figure 26c); in these data, as in the wet tests, no differentiation between the two wools is evident.

It is well known that lubrication has a profound effect on the performance of fabric in the flex abra-

[17] In the work quoted [17], the *dry* flex-abrasion resistance was compared with the *wet* fiber breaking energy.

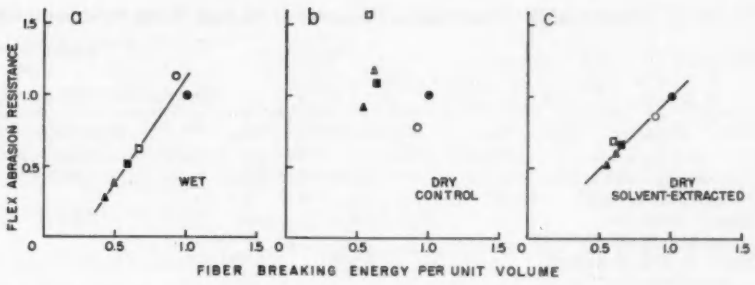

Fig. 26. Flex abrasion resistance as a function of fiber breaking energy. The symbols have the same meaning as in Figure 23. (Low-crimp ready-to-dye = 1.)

sion test; the present data offer a striking example of a case where lubrication has outweighed a substantial degradation in fiber properties.

Summary

The results of this study show that the fiber crimp present in raw wool largely persists through processing into a French-spun worsted fabric. The presence of the original fiber crimp in the fabric stage cannot be demonstrated by the methods used in this work, because of the superposition of fabric crimp onto it. Nevertheless, its effects on both subjectively evaluated and measurable fabric properties are evident in a structure unfavorable to the manifestation of crimp—relatively hard, fine yarn and a clear, tightly set fabric.

The comparison between the two wools differing in crimp content has shown that greater fiber crimp (1) increases the cohesion of twistless strands, and improves the spinning performance at the limit of the spin; (2) gives rise to a bulkier yarn with substantially lower extensional stiffness and breaking extension, and possibly a slightly lower breaking strength, in the normal range of twist; (3) results in a thicker fabric, with a softer handle and smoother appearance, greater air permeability, and possibly slightly lower bending stiffness and better drape; (4) yields an improvement in the recovery from extension and from wrinkling in the fabric (which is probably too small to be of practical significance); (5) appears to have no effect on the tensile properties of the fabric other than the initial stiffness in the dry state; and (6) has no influence on the resistance of the fabric to flex abrasion.

The two wools employed in the investigation have been found to differ in their sensitivity to acid damage, both in terms of alkali solubility and of fiber mechanical properties; the difference is revealed in dyeing, and consequently manifests itself in those fabric properties dependent on the fiber mechanical performance, such as resistance to flex abrasion.

Acknowledgments

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Some of the experimental work was performed by Donald J. Leitgeb, P. Cormac de MaCarty, and Louisa Templeton.

Appendix A

The Use of pH 9.2 Buffer in Testing

All the "wet" tests, on fibers and fabric, were performed in 0.1 *M* sodium borate, pH 9.2, 70° F., the material being immersed in the solution for 16–20 hr. prior to testing. The mildly alkaline solutions were chosen in order to increase the swelling and thus to emphasize the effect of any chemical degradation of the wool. There seems to be little likelihood

TABLE XVII. Fiber Tensile Properties of the High-Crimp Wool in pH 9.2 Borate Buffer at 70° F.

Property	Intact wool		Acid-treated wool		95% Limits of individual means
	Time in buffer				
	Overnight	7 da.	Overnight	7 da.	
Uncrimping stress, megagrams cm.$^{-2}$	0.035	0.037	0.019	0.020	±0.0029
Young's modulus, megagrams cm.$^{-2}$	13.4	13.1	8.5	9.0	±0.89
Stress at 20% extension, megagrams cm.$^{-2}$	0.459	0.463	0.277	0.283	±0.0098
Stress at break, megagrams cm.$^{-2}$	1.21	1.20	0.66	0.59	±0.069
Extension to break, %	51.4	50.3	48.6	42.9	±2.9
Energy to break per unit volume, megagrams cm.$^{-2}$	0.328	0.308	0.168	0.142	±0.024

that, at the pH and temperature used, the test solution itself will cause any damage, but the following experiment was performed to examine the possibility.

Intact fibers of the high-crimp wool and similar fibers which had been exposed to boiling 0.04 *N* sulfuric acid for 4 hr. were prepared for force-extension testing in the usual way and immersed in the buffer. Half the fibers (15 of each type) were extended to break after the normal overnight soaking, and the rest after 7 da. in the solution. The values for some of the properties derived from the stress-strain curve are summarized in Table XVII. Analyses of variance showed the "time in buffer" effect to be significant for breaking extension ($P = 0.02$) and nearly so for breaking energy ($P = 0.1$); for breaking extension, there is an indication that the effect is greater on the acid-treated than on the intact material (P for acid treatment × time in buffer = 0.1). No "time in buffer" effect was evident for any of the parameters of the stress-strain curve below the break.

The experiment is not completely conclusive, since there is a possibility that some changes occur within the first few hours of immersion in the buffer, without any subsequent alteration, but such a state of affairs seems unlikely. It may be assumed, therefore, that the pH 9.2 buffer at 70° F. does not itself produce any measurable changes in fiber mechanical properties, other than a slight loss in breaking extension and breaking energy on prolonged immersion.

Appendix B

Fiber Diameter and Assembly Bending Stiffness

The bending and torsional rigidity of a fiber is proportional to the fourth power of its diameter (assuming elastic moduli to be independent of diameter). Consequently, if the bending stiffness of a fiber assembly is dependent solely on the bending and torsional stiffness of its constituents, it will vary with the sum of the fourth power of the fiber diameter.

The average contribution of an individual fiber will then be proportional to the mean of the fourth power of diameter ($\overline{x^4}$). The values of this parameter for the two wools under consideration are: low-crimp, 3.382×10^5; high-crimp, 2.950×10^5 microns^4, their ratio being 1.146.[18]

This ratio represents the fourth-power-of-diameter effect for the two assemblies containing the *same number* of fibers.

In *assemblies of the same weight,* the number of fibers present is inversely proportional to the mean second power of diameter ($\overline{x^2}$), which is 5.235×10^2 for the low-crimp, and 5.056×10^2 microns^2 for the high-crimp wool. The ratio for the two assemblies of the same weight, $(\overline{x_L^4}/\overline{x_H^4})$ $(\overline{x_H^2}/\overline{x_L^2})$, is 1.107. Thus, a 10.7% difference between as-

[18] For a Gaussian distribution, the mean of the fourth power is equivalent to the fourth power of the arithmetic mean multiplied by the fourth moment [25]:

$$\overline{x^4} = \frac{\Sigma x^4}{n} = (\bar{x})^4\left[1 + 6\left(\frac{s}{\bar{x}}\right)^2 + 3\left(\frac{s}{\bar{x}}\right)^4\right]$$

In the present instance, this equation yields a value of 1.080 for the low/high ratio; the difference between this and 1.146 is due to the departure of the distributions from normality.

semblies of the same weight will manifest itself in any property dependent on the fourth power of fiber diameter, all else being equal.

In fact, no assembly property will have such a high diameter-dependence. Fabric bending, for example, will be influenced by fiber extensional properties and interfiber friction as well as by fiber bending. The ratio 1.107, therefore, represents an upper limit of the diameter effect.

It may therefore be concluded, conservatively, that any fabric property in which a ratio (low crimp/high crimp) greater than 1.107, or smaller than unity, is observed must contain a contribution from factors other than fiber diameter. For the intermediate range, a separation of the effects is impossible.

Literature Cited

1. Alexander, E., Lewin, M., Musham, H. V., and Shiloh, M., TEXTILE RESEARCH JOURNAL **26**, 606 (1956); **26**, 803 (1956).
2. American Society for Testing Materials, Tentative Specifications and Method of Test for Fineness of Wool (D419–55T); Tentative Specifications and Method for Test for Fineness of Wool Tops (D472–55T).
3. Anderson, S. L., *J. Textile Inst.* **45**, P312 (1954); **47**, P960 (1956).
4. Anon., *Wool Science Review* **8**, 33 (1952).
5. Anon., *Wool Science Review* **11**, 40 (1953).
6. Brown, A., TEXTILE RESEARCH JOURNAL **25**, 969 (1955).
7. Burte, H. M., TEXTILE RESEARCH JOURNAL **23**, 469 (1953).
8. Burte, H. M., TEXTILE RESEARCH JOURNAL **24**, 715, 726 (1954).
9. Chaikin, M., Baird, K., Stutter, A. G., and Curtis, J., *Proc. Int. Wool Text. Res. Conf. Austr.* 1955, **E**, 168.
10. Chu, C. C., Cummings, C. L., and Teixeira, N. A., TEXTILE RESEARCH JOURNAL **20**, 539 (1950).
11. deMaCarty, P. C., and Dusenbury, J. H., TEXTILE RESEARCH JOURNAL **25**, 875 (1955).
12. Dillon, J. H., *Ind. Eng. Chem.* **44**, 2115 (1952).
13. Duerden, J. E., *J. Textile Inst.* **20**, T93 (1929).
14. Dusenbury, J. H., unpublished results.
15. Dusenbury, J. H., and Jeffries, E. B., *J. Soc. Cosmetic Chem.* **6**, 355 (1955).
16. Dusenbury, J. H., and Menkart, J., *Proc. Int. Wool Text. Res. Conf. Austr.* 1955, **F**, 142.
17. Dusenbury, J. H., Mercer, E. H., and Wakelin, J. H., TEXTILE RESEARCH JOURNAL **24**, 890 (1954).
18. Evans, T. F., TEXTILE RESEARCH JOURNAL **24**, 637 (1954).
19. Evans, T. F., and Montgomery, D. J., TEXTILE RESEARCH JOURNAL **23**, 689 (1953).
20. Felix, E., *Textil-Rundschau* **10**, 1 (1955).
21. Goldworthy, Y. E., and Lang, W. R., *J. Textile Inst.* **45**, T755 (1954).
22. Hamburger, W. J., private communication.
23. Hamburger, W. J., TEXTILE RESEARCH JOURNAL **15**, 169 (1945).
24. Horio, M., and Kondo, T., TEXTILE RESEARCH JOURNAL **23**, 373 (1953).
25. Kendall, M. G., *The Advanced Theory of Statistics*, Griffin & Co., London (1947).
26. Lang, W. R., *J. Textile Inst.* **38**, T241 (1947).
27. Lang, W. R., *J. Textile Inst.* **38**, T257 (1947).
28. Lang, W. R., and Skertchly, *J. Textile Inst.* **46**, T435 (1955).
29. Menkart, J., TEXTILE RESEARCH JOURNAL **27**, 186 (1957).
30. Mercer, E. H., TEXTILE RESEARCH JOURNAL **23**, 388 (1953); **24**, 39 (1954).
31. Montgomery, D. J., and Milloway, W. T., TEXTILE RESEARCH JOURNAL **22**, 727 (1952).
32. O'Connell, R. A., and Yeiser, A. S., TEXTILE RESEARCH JOURNAL **24**, 629 (1954).
33. Rainard, L. W., and Abbott, D., TEXTILE RESEARCH JOURNAL **20**, 301 (1950); Barach, J. L., and Rainard, L., *ibid.* **20**, 308 (1950).
34. Roberts, N. F., private communication.
35. Roberts, N. F., TEXTILE RESEARCH JOURNAL **26**, 687 (1956).
36. Roberts, N. F., and Dunlop, R. A., paper presented at the Int. Wool Text. Res. Conf., Austr. (1955).
37. Simmonds, D. H., private communication.
38. Thorsen, W. J., private communication.
39. U.S. Department of Agriculture, Amendment of Regulations of the Secretary of Agriculture Relating to the Official Standards of the United States for Wool (Nov. 1942).
40. von Bergen, W., private communication.
41. von Bergen, W., and Wakelin, J. H., TEXTILE RESEARCH JOURNAL **22**, 123 (1952).
42. Wakelin, J. H., Lambert, H. W., and Montgomery, D. D., TEXTILE RESEARCH JOURNAL **26**, 665 (1956).
43. Whewell, C. S., *J. Textile Inst.* **47**, P851 (1956).
44. Whitwell, J. C., and Menkart, J., unpublished results.
45. Wilson, L. T., *Austr. J. Agric. Res.* **2**, 195 (1951).
46. Woods, H. J., *J. Textile Inst.* **26**, T93 (1935).
47. Young, S. S. Y., and Dunlop, A. A., paper presented at the Int. Wool Text. Res. Conf., Austr. (1955).
48. Zahn, H., *Kolloid Z.* **113**, 157 (1949); *Textil-Praxis* **5**, 315 (1950).

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The Elastic Properties of Chemically Modified Wools and Their Implications for Theories of Wool Structure

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Abstract

The changes in elastic properties produced by systematic changes of the number and kind of sulfur bonds are interpreted as implying the existence in the wool fiber of units joined longitudinally by disulfide bonds. Some possible implications of this concept are discussed.

RECENT observations from these laboratories have suggested that soluble proteins derived from wool by either oxidation or reduction procedures are cyclic peptides [8, 32] which have a minimum molecular weight, as low as 8–10,000 [15]. Since in the case of the oxidation procedures the fraction obtained amounts to 90% of the weight of the wool [2], and 70% in the case of reductive procedures [12], it would seem that a re-assessment of theories of wool structure is required. From work on the relation of fiber strength to the molecular weight of the fundamental unit [23], it has been suggested that a minimum molecular weight of 10,000 is required to account for the strength of the wool fiber [22]. However, this estimate is derived from data on open-chain β structures, and probably a higher value would be required for cyclic α structures. Some form of end-to-end aggregation seems to be a necessary postulate. A similar conclusion is necessary to account for the low angle meridional X-ray diffraction photograph of wool which implies considerable structural regularity in the direction of the fiber axis extending for considerable distances [6]. Since the evidence rules out peptide bond fission during the isolation of the proteins from wool [8, 32], from the chemical nature of the techniques used to isolate them, the simplest conclusion which can be drawn is that in the fiber these units are joined end to end through disulfide bonds, as has been proposed earlier [19]. It is difficult at present to see how direct evidence for this suggestion can be obtained, but it has long been a tenet of polymer chemistry that macroscopic elastic properties can be related to molecular structure. Accordingly, an investigation of the alteration in elastic properties of wool fibers produced by systematic variation of the number and kind of disulfide bonds [25] was undertaken, and this paper records the results.

Experimental

The wool used throughout was a Lincoln 36's in the staple form cleaned by cold extraction with ethanol (3X) followed by rinsing in several changes of distilled water. Fiber bundles of approximately 0.1 g. and 10 cm. length were combed out from the staples and had their ends set in blocks of Epikote resin (Shell chemicals). All treatments were carried out on such bundles, the object of the technique being to prevent fiber felting during treatment. After treatment the blocks were cut off and approximately 5-cm. lengths were attached to glass hooks for determination of load-extension curves on the recording Cambridge extensometer at constant rate of loading (2.3 g./min.).

Table I lists the type of chemical treatment and its effect on the cystine links. The nomenclature A, B, C, and D for the cystine fractions is that of Middlebrook and Phillips [25].

The following comments provide additional information to Table I. The occurrence of djenkolic acid and dimethylene SS′ bis cysteine in hydrolysates of the wool after treatments (iii) and (v) was confirmed by paper chromatography. Direct evidence for the existence of the sulfone of dimethylene SS′ bis cysteine in hydrolysates of wool after treatment (vi) could not be obtained. However, the com-

pound was prepared by oxidation of dimethylene SS′ bis cysteine with 1% peracetic acid overnight. It was a white crystalline solid, m.p. 206° (uncorr.), not very soluble in water but soluble in acid or alkaline solution. Analysis showed 28.5% C, 4.9% H, 37.7% O, 19.0% S. Calculated values were 28.9% $C_8H_{16}N_2S_2O_8$:C, 4.9% H, 38.5% O, and 19.3% S. On heating in a sealed tube overnight with 6 *N* HCl at 110°, it decomposed completely to give at least five ninhydrin positive materials on a paper chromatogram, the two major components being probably alanine and cysteic acid. Failure to detect the amino acid in the hydrolysate of wool after treatment (v) is therefore understandable.

In view of the importance attached later to the load-extension curves obtained from fibers which had received treatment (vi), indirect evidence was sought that the dimethylene SS′ bis cysteine linkages had survived the subsequent peracetic acid oxidation. The solubility in 1 *N* ammonia solution after 24 hr. of a sample treated in this way was compared with the solubility of peracetic treated wool. The respective solubilities were 50 and 90%, confirming the existence of cross-links in the sample. More impressive than these figures, however, was the behavior of the two samples on immersing in the ammonia solution. Whereas the wool which had been merely oxidized with peracetic acid immediately swelled to a gelatinous mass, the wool which had been cross-linked prior to oxidation stayed comparatively unswollen for several hours, and it seemed probable that the swelling and solution which eventually took place were subsequent to rupture of the sulfur cross-links by the alkali. Other evidence relating to this point is discussed later.

None of the treatments used caused any changes in length of the fibers except (vii), which always caused supercontraction, usually of the order of 15–20%, but occasionally as much as 30%. This is of importance since extensions considerably in excess of 100% have been quoted [1] for such fibers as evidence against the claim by Astbury and Woods [5] that the limiting extension of wool is around 100%. It can readily be seen that if a fiber has supercontracted by 30%, an extension of 200% on this supercontracted length corresponds to only 110% extension on the original length. In the present work, extensions were calculated on the original length and the maximum extension at break observed was 107%.

Results

Figure 1 shows typical load-extension curves of fibers after treatments (i–ix) in Table I. Treatments (ii) and (iii) were indistinguishable in their effects on the fiber. It can be seen that the results parallel closely the earlier ones of Harris and Brown [17]. Correlation of the results with the A, B, C, D fractions of Middlebrook and Phillips [25] produces some interesting observations. Thus it appears that it is a fraction A + B which is largely responsible for limiting the ultimate extension of wool to 70% or less. This increase in ultimate extensibility with reduction of the disulfide bonds pro-

TABLE I

	Treatment of fiber	Effect on cystine bonds
(i)	Untreated	All intact (SS = 2.59% S).
(ii)	20% $NaHSO_3$ solution 24 hr.; wash in distilled water overnight.	Subfraction B broken; A, C, and D intact [25] (SH = 0.49% S).
(iii)	Reduced 0.5 *M* sodium thioglycollate pH 5 overnight, cross-linked methylene iodide pH 8.5 buffer 24 hr., washed in water.	Subfraction B as SH, subfraction A cross-linked as djenkolic acid. C + D intact [26].
(iv)	Reduced 0.5 *M* sodium thioglycollate pH 5 overnight, washed in water.	A + B broken (as SH); C + D intact [25] (SH = 1.26%).
(v)	As (iv) but subsequently cross-linked with ethylene dibromide pH 8.5 overnight.	A + B cross-linked as dimethylene SS′ bis cysteine, C + D intact [26].
(vi)	As (v) but subsequently oxidized with 1.6% peracetic acid overnight [2].	C + D broken (as cysteic acid); A + B, presumably sulfone of dimethylene SS′ bis cysteine.
(vii)	Untreated wool oxidized with 1.6% peracetic acid overnight [2].	>90% broken by conversion to cysteic acid.
(viii)	As (v) but subsequently reduced 0.5 *M* thioglycollate pH 10.7 and recross-linked with ethylene dibromide.	A + B and large proportion of C + D converted to dimethylene SS′ bis cysteine (SS = 0.44% S).
(ix)	As (viii) then oxidized with 1.6% peracetic acid overnight.	Mainly cross-linked as sulfone of dimethylene SS′ bis cysteine.

vides a strong argument against the possibility that extension of the wool fiber is accompanied by rupture of disulfide bonds.

The most interesting result, however, is shown by fibers which have been oxidized subsequent to cross-linking A + B with bis thioether linkages. Although cross-linking alone produces a fiber very similar in properties to the original wool, subsequent oxidation produces a fiber which is extremely weak and in fact is very little stronger than a fiber which has had all its cystine links destroyed by peracetic acid (vii). In these circumstances, it is important to establish whether or not the bis thioether cross-linkages have been destroyed by the peracetic acid. As has already been mentioned, direct confirmation, by demonstrating the occurrence of the sulfone of dimethylene SS′ bis cysteine in the hydrolysate of treated wool, is impossible because of its instability to acid hydrolysis. Circumstantial evidence for the persistence of cross-links in the isolated wool after peracetic acid oxidation is quite strong, however, viz.:

1. The free dimethylene SS′ bis cysteine reacts with peracetic acid to give the sulfone.
2. The solubility in 1 *N* ammonia of the wool presumed to contain the sulfone is considerably less than that of wool in which no cross-links and only cysteic acid is present.
3. The cross-linked wool, unlike the untreated wool, does not supercontract during subsequent oxidation with peracetic acid.
4. The limiting extension of the fibers is around 75%, whereas if the peracetic acid oxidation had broken the bis thioether linkages an ultimate extensibility of 100% could be expected.

If the conclusion is accepted that the bis thioether cross-links of the A + B fraction have not broken during the subsequent oxidation, then the conclusion immediately follows, from the results shown in Figure 1, that the C + D cystine linkages are more fundamentally important in wool structure than the A + B. The latter contribute considerably to the strength of the intact fiber, but are relatively ineffective if the C + D bonds are ruptured.

The results of load-extension curves on fibers after treatments (viii) and (ix) (Figure 1) furnish some further confirmation of the great difference in structural significance of the A + B and C + D links. Originally these treatments were envisaged mainly as controls on the effect of oxidation on the bis thioether linkages, since if all the cystine linkages can be replaced by cross-linkages of this kind, it should be very simple to detect rupture of them by subsequent oxidation. The curves do in fact show that oxidation produces no loss of ultimate breaking strength of the fibers, although the oxidized fibers apparently extend further. The most striking feature of the curves, however, is the great loss in strength produced by the second reduction and coupling at pH 10.7. Whereas the first pH 5 reduction and coupling produces comparatively little change in the elastic properties of the wool (cf. (i) and (v), Figure 1), the second treatment has caused a large drop in breaking load. It would seem that when wool is reduced at pH 5, subsequent coupling with ethylene dibromide either couples the same sulfur atoms together which formed the original A + B disulfide bonds to give structurally similar links or that the precise replication of the original structure is unimportant. When the C + D links are broken by reduction at pH 10.7, however, it appears that the original linkages are not reformed by subsequent treatment with ethylene dibromide, thus emphasizing from another viewpoint the different structural importance to be attached to the C + D fraction of the cystine.

Discussion

The simplest assumption to account for the great loss in strength of wool fibers when the S bonds of the C + D fractions of the cystine are ruptured is that the average chain length has been considerably reduced, i.e., that the keratin molecules are joined end to end and through disulfide bonds. Since the evidence is strongly against any of the cystine of wool being incorporated in any fashion which leaves unbonded either carboxyl or amino groups [19, 27], this conclusion implies the occurrence of cyclic structures as envisaged earlier by Lindley and Phillips [19]. The conclusions by Thompson [32] and Bradbury [8] that wool may consist largely of cyclic proteins thus agrees well with this picture. This end-to-end aggregation through sulfur bond formation to give fibrils also fits in extremely well with the views expressed on biogenesis of the wool fiber, as revealed by recent electron microscope investigations [24].

If this view be accepted, the interesting question remains as to the size of the fundamental units.

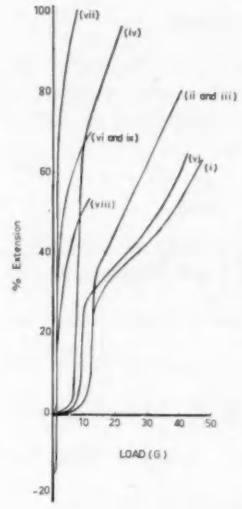

Fig. 1. Load vs. elongation, treated wools.

Values for the molecular weight of soluble wool keratin derivatives varying from 8,000 to 80,000 have been reported by various workers. There is little doubt, however, that these soluble keratin derivatives aggregate extremely readily, and it may be significant that Harrap's value of 8–10,000 [15] was obtained using surface balance techniques, which permit lower protein concentrations to be used than are possible in conventional sedimentation, electrophoresis, diffusion, or osmotic techniques. The surface balance technique may therefore lead to disaggregations which are difficult to observe by other methods and could give a more reliable value for minimum molecular weight. This effect has already been observed in the case of insulin, where the surface balance technique gives the value of 6,000 [16]. Other extremely significant evidence in favor of the value given by the surface balance technique, as being nearer the true value of the minimum molecular weight, is furnished by data on horse hemoglobin. The usual hydrodynamic methods give a molecular weight of about 68,000 for this protein, and Porter and Sanger [30] found six terminal valine end groups per molecule. Ingram [18] found that there were six cysteine residues per molecule and no cystine, so that unless some novel method of cross-linking is invoked, the molecule consists of six peptide chains held together by secondary valence forces only. However, even in 8 *M* urea solution splitting occurs to half molecules only [13, 31], and no evidence exists from bulk solution measurements of any breakage to units smaller than this. Guastalla [14] in 1939 (i.e., long before there was any evidence regarding the number of chains in the hemoglobin molecule) showed by the surface balance method that horse hemoglobin has a molecular weight of 12,000. There is thus strong presumptive evidence that horse haemoglobin can be dissociated at the surface into its individual peptide chains, whereas in bulk solution there is no evidence for anything smaller than groups of three chains. The derived wool proteins may well have similar properties; indeed, to fulfill their function they must be strongly aggregating, and if so, surface balance techniques may furnish the only reliable method of obtaining the minimum molecular weight, especially if the chains are cyclic.

It is worth noting that the low value for the ultimate strength of fibers which have the A + B cystine cross-linked but the C + D cystine broken [treatment (vi)] is good evidence that the mean chain length must be quite short. The protein chains are still extensively covalently cross-linked, yet the fibers are much weaker than regenerated protein fibers, suggesting that the mean chain length of the keratin molecules is very short.

A molecular weight of 9,000 corresponds to about 80 amino acid residues in the case of wool; hence the maximum length of a cyclic molecule of this size in the α configuration should be $(80/2) \times 1.5 = 60$Å. Attaching a cystine link at each end could add approximately another 9Å to the length of the unit. This is in reasonable agreement with the 66Å spacing observed in the X-ray picture, but conflicts with the 100Å particle size claimed from electron microscope studies [10]. However, at present there is a completely unresolved conflict between interpretation of electron micrographs and X-ray data at this level of organization. It would seem very difficult to reconcile these ideas with the suggestion of a seven strand cable proposed by Pauling and Corey [28], and in any case the density calculated from this structure seems to be low [11]. In order to account for the 5.1Å meridional spacing, it probably would be necessary to retain the suggestion of "coiled coils" [9, 28], and the cyclic molecules accordingly would form two strand cables. Since the X-ray data cannot be indexed on a meridional spacing of less than 198Å (i.e., 3×66), it would seem possible that this either represents the

pitch of the cable and 3 units of 66Å are required to give a true repeat, or that each 66Å unit is rotated 120° with respect to its neighbors along the fiber axis. Fraser and MacRae propose to discuss some of these aspects of keratin structure in detail in a forthcoming publication.

Astbury's assumption that α keratin is transformed to β keratin when a wool fiber is stretched is open to some doubt [7, 10], and is almost certainly not compatible with the Pauling and Corey structure [20, 21]. If Astbury's assumption be accepted, the suggestion of cyclic molecules fits in well with the observation that the β photograph of stretched wool requires two anti-parallel chains as the structural unit [3, 4]. On the other hand, the meridional spacing of 3.3Å on the β photograph of stretched wool is in much better agreement with the parallel pleated sheet of Pauling and Corey [29] than with their anti-parallel pleated sheet. Thus this observation also constitutes the strongest single piece of evidence against the hypothesis of cyclic structures, since it is difficult to see how cyclic units can be reconciled with a parallel chain structure. However, it is possible that neither the parallel or anti-parallel pleated sheet is the correct model for stretched wool. Thus the cyclic units could be arranged in such a way as to give alternate pairs of parallel and anti-parallel chains. A β structure derived from this would be different from either of the Pauling and Corey models. Rough model building experiments suggest that a β structure of this type may be possible; further, more accurate investigation is planned.

The theoretical extensibility of an α-helix should be 122% on transformation to the β configuration, i.e., $[(3.3/1.5) - 1]\ 100$, but on the present model the presence of inextensible disulfide bonds between the cyclic units would restrict this to a lower value. The calculated extensibility of a 66Å unit containing a 9Å disulfide bond is about 105%; this agreement could of course be fortuitous.

Although it is realized that many of the points raised in the foregoing discussion are extremely speculative, nevertheless it is felt that the underlying assumption of fairly small cyclic protein molecules joined end to end through disulfide bonds provides the most satisfactory general picture of keratin structure yet proposed.

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Literature Cited

1. Alexander, P., *Proc. Roy. Soc. Med.* **44**, 389 (1951).
2. Alexander, P., Fox, M., and Hudson, R. F., *Biochem. J.* **49**, 129 (1951).
3. Astbury, W. T., *Proc. Int. Wool Textile Res. Conf. Australia* **B**, 202 (1955).
4. Astbury, W. T., and Street, A., *Phil. Trans. Roy. Soc.* **A230**, 75 (1931).
5. Astbury, W. T., and Woods, H., *Phil. Trans. Roy. Soc.* **A232**, 333 (1933).
6. Bear, R. S., and Rugo, H. J., *Ann. N. Y. Acad. Sci.* **53**, 461 (1951).
7. Bendit, E. G., *Nature* **179**, 535 (1957).
8. Bradbury, J. H., submitted to *Biochem. J.*
9. Crick, F. H. C., *Nature* **170**, 882 (1952).
10. Farrant, J. L., Rees, A. L. G., and Mercer, E. H., *Nature* **159**, 535 (1947).
11. Fraser, R. D. B., and MacRae, T. P., TEXTILE RESEARCH JOURNAL **27**, 384 (1957).
12. Gillespie, J. M., and Lennox, F. G., *Aust. J. Biol. Sci.* **8**, 97 (1955).
13. Greenberg, D. M., and Burk, N. F., *J. Biol. Chem.* **87**, 197 (1930).
14. Guastalla, J., *Compt. Rend.* **208**, 1078 (1939).
15. Harrap, B. S., *Proc. Int. Wool Textile Res. Conf. Australia* **B**, 86 (1955).
16. Harrap, B. S., *J. Colloid. Sci.* **10**, 351 (1955).
17. Harris, M., and Brown, A. E., *Symp. Fibrous Proteins*, Soc. Dyers Colourists, Bradford, 203 (1946).
18. Ingram, V. M., *Biochem. J.* **59**, 653 (1955).
19. Lindley, H., and Phillips, H., *Biochem. J.* **39**, 17 (1945).
20. Lindley, H., *Proc. Inter. Wool Textile Res. Conf. Australia* **F**, 120 (1955).
21. Low, B. W., *The Proteins* **1**, 383, Academic Press Inc., New York (1955).
22. Lundgren, H. P., *Adv. Protein Chem.* **5**, 305 (1949).
23. Mark, H., *Indus. Eng. Chem.* **34**, 1343 (1942).
24. Mercer, E. H., Farrant, J. L., and Rees, A. L. G., *Proc. Int. Wool Textile Res. Conf.* **F**, 120 (1955).
25. Middlebrook, W. R., and Phillips, H., *Biochem. J.* **36**, 428 (1942).
26. Middlebrook, W. R., and Phillips, H., *Biochem. J.* **41**, 218 (1947).
27. Patterson, W. L., Geiger, W. B., Mizell, L. R., and Harris, M., *Bur. Stand. J. Res.*, Wash., **27**, 89 (1941).
28. Pauling, L., and Corey, R. B., *Nature* **171**, 59 (1953).

29. Pauling, L., and Corey, R. B., *Proc. Nat. Acad. Sci.* **39**, 247 (1953).
30. Porter, R. R., and Sanger, F., *Biochem. J.* **42**, 287 (1948).
31. Steinhardt, J., *J. Biol. Chem.* **123**, 543 (1938).
32. Thompson, E. O. P., *Aust. J. Biol. Sci.* **10**, 225 (1957).

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A Method for Studying the Deformation of Fabrics in Creasing

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Abstract

A procedure has been devised for obtaining the load-deformation curve of a fabric during a deformation which begins as a small-radius bend and ends in crease formation. A strip of the fabric is formed into a cylinder and mounted horizontally on the crosshead of an Instron tester. A fixed weight is hung from the strain gauge and the force exerted on the bottom of this weight as the sample cylinder moves up against it is recorded by the tester. The recovery curve can be obtained by reversing the crosshead. The results are most conveniently presented by plotting log of the jaw separation against the reciprocal of the load. Curves obtained in this way on a variety of fabrics, including some resin-finished ones, indicate that the procedure should be of value in studying stiffness, resilience, crease recovery, and other properties related to the drape and handle of fabrics.

THE flexural behavior of a fabric at small bending radii is important in its handle and draping properties. When the bending radius is of the order of the fabric thickness, the result is creasing and wrinkling, which can play a large role in the appearance and utility of the material, particularly in clothing. Fabric characteristics in this respect are usually evaluated in the laboratory by crease recovery and stiffness tests. The usual crease recovery measurement is a determination of recovery at a fixed time after a fabric strip has been creased and unloaded, and gives no indication of the behavior of the fabric while it is being creased. Fabric stiffness is usually evaluated by the cantilever test [1] or some other procedure which enables the flexural rigidity to be calculated. The actual deformation in the test usually depends on the stiffness itself, but it is frequently quite small compared with that involved in creasing. Neither test can be used to describe adequately the load-deformation behavior of fabrics when the bending radius is varied in the range from about 1 cm. down to the thickness of the fabric, as it is when a crease is formed. This paper describes a new procedure for studying fabric bending in this range and shows how it characterizes the fabric's behavior more completely than can be done by crease recovery and stiffness measurements alone.

Earlier workers have recognized the importance of measurements of this sort and have devised various techniques for making them. McNicholas and Hedrick [5], who were interested in the speed with which parachutes unfolded and opened, measured load-deformation curves for folded cloth samples using a manual technique and a simple balance. The folded sample was raised on a platform under a weight which was counterpoised on one arm of the balance, and then the weight necessary to rebalance the beam was determined. The deformation involved was measured on the rack and pinion device for lifting the sample platform.

Instruments for making measurements of this type have also been developed by Schiefer [6], Zyrin [7], and Finch [3]. The procedure described below uses a sample arrangement somewhat similar to that

of Zyrin, and the Instron tester which was used here is in many respects like the instrument used by Finch. The present technique is different from these earlier ones, and by use of new load-deformation plot the results over a wider range of these parameters can be conveniently displayed.

Experimental Procedure

The procedure is carried out on an Instron model TT-B tensile tester [4] using a simple arrangement which in effect converts it into a compression tester for light loads.

A strip 1.5 cm. wide by 7.5 cm. long is cut from the fabric to be tested and mounted on a 1 × 3-in. microscope slide by butting the ends together in a thin drop of Duco cement on the slide. The sample then forms a circle about 1 in. in diameter, although unless the fabric is fairly stiff it will sag under its own weight. The drop of cement should be small enough to avoid gluing an appreciable length of the fabric flat to the glass and distorting the circular form. The mounted sample is then placed on a small horizontal platform on the crosshead of the Instron tensile tester, as shown in Figure 1. The tester is in a room maintained at the standard temperature and humidity for textile testing. A 1-lb. weight is hung on the strain gauge with another microscope slide cemented to its bottom. This slide must be carefully centered and the weight carefully suspended so that when the two slides are brought together by raising the crosshead they are quite parallel as they approach one another. This is best checked with a clear slide in the sample position by slowly bringing the slides very close to each other with the manual control.

The load-measuring section of the tester is set up and calibrated in the following way so that the increasing load borne by the sample registers as a left-to-right motion of the pen. The "B" cell strain gauge is used with the leads to the bridge amplifier reversed so that the motion of the recorder pen is from right to left as the strain gauge is loaded. With the amplifier on high sensitivity and with the 1-lb. weight on the strain gauge, the pen is adjusted to zero with the balance controls. The beam is raised to lift the weight, the range switch is moved to 50, and the pen set at some point near its maximum movement to the right with the calibration control. It would be best if this point could be made to be 10 on the scale of the paper, but on the tester used here range of the calibration control limited the pen setting to 7.4. This arrangement produces the maximum spread of the record on the chart paper. The scale calibration factor is not very convenient, but since the chart record is replotted as described below, it is fairly simple to take readings from the chart at regular scale readings for which the load has been previously calculated.

An actual test is carried out in the following manner. The mounted sample, after being allowed to condition again after it was mounted, is placed on the crosshead and brought up close to the bottom of the weight with the manual crosshead control. With the range switch on 1 and a chart speed of 5 in./min., the crosshead drive is started up at 0.1 in./min. As the pen approaches 2 on the scale (2.40 g.), the range switch is changed to 2. When the pen reaches 8 or 9 on range 2 (about 20 g.), the range is switched to 50. In this position the pen will read 7.4 when the maximum load is applied by lifting the weight from the hook with the sample. The occurrence of the latter is very apparent from the record, and the crosshead is then stopped. After 10 sec. the crosshead is started downward and the recovery curve is determined, range changes being made at the same places as before. The time that the weight rests on the sample before the crosshead is started down is not very critical. It was found that increasing the

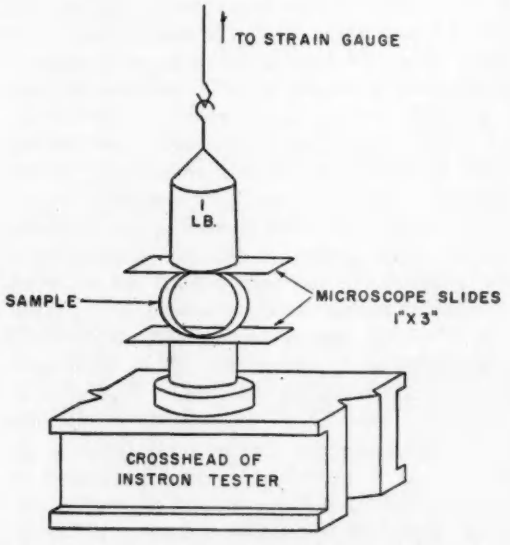


Fig. 1. Arrangement for determining load-deformation curves for fabric creasing.

time to 5 min. made no significant change in the recovery.

When the sample no longer touches the weight, the slide bearing it is removed, cleaned with acetone, and replaced. The crosshead is started up again and a blank run made to indicate the zero jaw separation point on the chart. The slide is allowed to lift the weight, and the crosshead is reversed. The cross-chart lines produced by lifting and rehanging the weight are not identical but should be quite close together. The average of the two is taken as zero jaw separation. If the two lines are not close, it usually indicates that the suspension of the weight is not quite balanced, either because of a bent hook, improper centering of the upper glass slide, or unlevelness of the tester itself.

The load varies from a few tenths of a gram soon after the contact is made between the weight and sample to 460 g. (the weight of the pound weight plus the glass slide). Plotting such a range of loads against deformation (which is measured by the jaw separation d) is not very convenient or meaningful on straight Cartesian coordinates. On a strictly empirical, trial-and-error basis, we have found that an easy way to handle the data is to plot deformation against the reciprocal of the load on log-log paper. There is no theoretical basis for doing this.

A typical plot is shown in Figure 2. The test begins at the point A. The distance between the curve and the 1 in. scale mark on the jaw separation axis (indicated by the arrow) is a measure of how much the sample ring of fabric has sagged under its own weight. As the test proceeds, the load increases (moving left along the curve) until the minimum jaw separation is reached at B. The shape of the curve between A and B is characteristic of the small-radius bending behavior of the test piece. The jaw separation at B is equal to the thickness of a double layer of fabric and can be checked with dial-gauge measurements. When the crosshead is reversed the recovery of the sample is determined by the lower member of the curve which begins leveling off near point C. The distance on the deformation scale between B and C is related to the crease recovery of the fabric as determined by the conventional tests. For a quantitative estimate of the ultimate recovery of the sample, the ratio of the value on the recovery curve at $1/L = 10$ to the corresponding point on the deformation curve is used.

Some Applications of the Procedure

Various Fibers and Fabrics

A variety of fabrics of different constructions and fiber contents were tested and the results for some fabrics showing various types of behavior which were observed are shown in Figures 3 and 4.

The upper curve in Figure 3 was obtained on a linen crash. This was a relatively stiff fabric, and when the sample ring was formed, it sagged only very slightly under its own weight. The arrow in the figure indicates the diameter which the undeformed ring would have and points toward the part of the curve where the test began. As the jaw separation becomes smaller, the load at first rises relatively rapidly. The curve then turns downward in a region which has the appearance of a yield point in tensile behavior. Then it more slowly approaches the final deformation value of twice the fabric thickness. The recovery curve is considerably lower than the deformation curve and levels off when the sample ring is about 36% as high as it was when the test began. The stiff behavior of the fabric early in the test and the very poor recovery are recognizable as characteristics usually associated with this type of fabric.

The second curve in Figure 3 is for an acetate satin and its general shape is much like that of the curve for linen. The sample deforms a little more under its own weight and is considerably less stiff, the "yield region" of the curve appearing at a lower load. The fabric is considerably thinner than the linen, and though the recovery curve is considerably below the deformation curve, the ultimate recovery (60%) is quite a bit higher than that of linen.

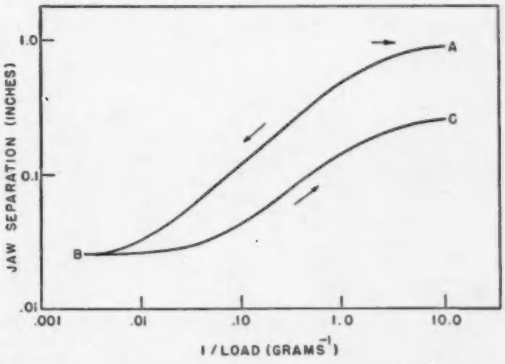


Fig. 2. Typical load-deformation curve for fabric creasing.

The lowest curve in Figure 3 is for an unfinished spun nylon dress fabric. This fabric is very limp, drooping heavily under its own weight and showing no sign of stiffness in the deformation curve. It is almost the same thickness as the acetate satin. The recovery curve is somewhat closer to the deformation curve than for the acetate, but the ultimate recovery is very poor (28%).

The top curve in Figure 4 is for a wool flannel. It supports its own weight fairly well but shows very little stiffness. The recovery curve is close to the deformation curve, and the ultimate recovery is 60%. It is obvious that this fabric is quite thick, and this also contributes to the lower incidence of sharp creases and wrinkles which impair appearance. The second curve in Figure 4 is for a blended viscose-wool gabardine. It, like the flannel, shows good recovery and reaches an ultimate value of 67%. The bottom graph is a curve for cotton print cloth. It shows little stiffness, and its recovery is intermediate between the linen and the flannel, reaching an ultimate value of 44%.

It is apparent from these examples that the curves obtained from various fabrics are quite distinctive and have features which may be related to their useful properties.

Cyclic Deformation

The creasing load-deformation behavior of cotton sheeting during repeated cyclic deformation is shown in Figure 5. In the upper curve the crosshead was reversed as loads of 1, 10, 100, and 460 g. were

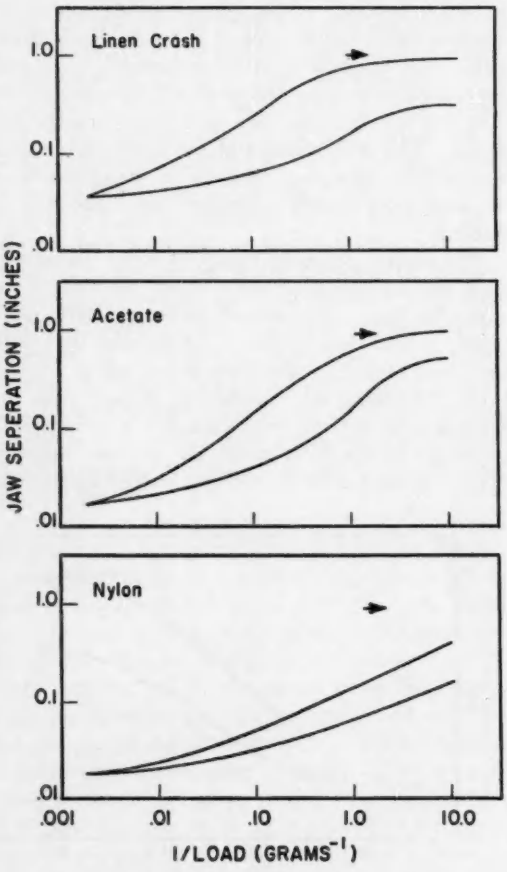


Fig. 3. Creasing load-deformation curves for various fabrics.

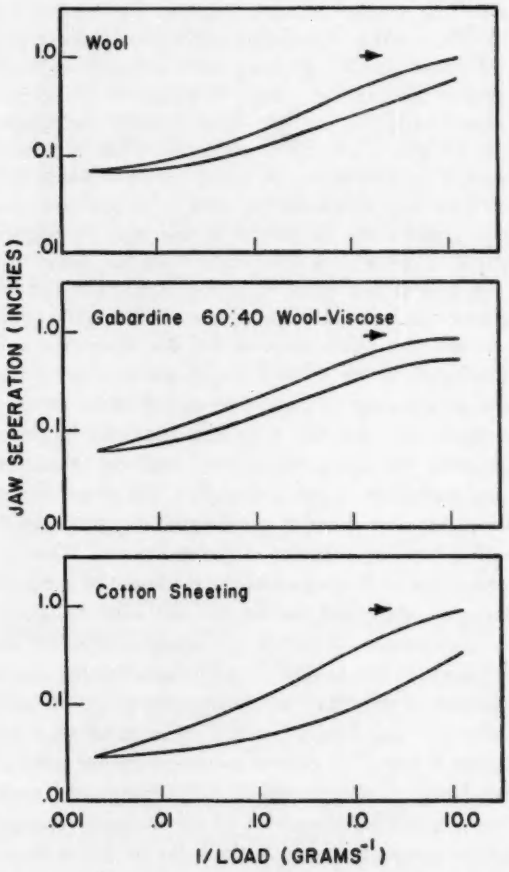


Fig. 4. Creasing load-deformation curves for various fabrics.

reached in successive cycles. The lower limit of each cycle was a load of 0.1 g. The upper envelope formed by the successive deformation curves is essentially the same as the curve obtained by a single deformation to the highest load. However, the ultimate recovery of the sample has been found in general to be somewhat lower than for the single cycle test.

A cycling test in which each cycle is carried to the maximum load is shown in the lower part of Figure 5. The curves for the first, second, and fifth deformation cycles on cotton print cloth are shown. Most of the irreversible deformation occurs in the first cycle, and change in the succeeding cycles is relatively slow.

The results of both kinds of cyclic tests are very similar to those obtained in other tests involving repeated deformation of viscoelastic materials.

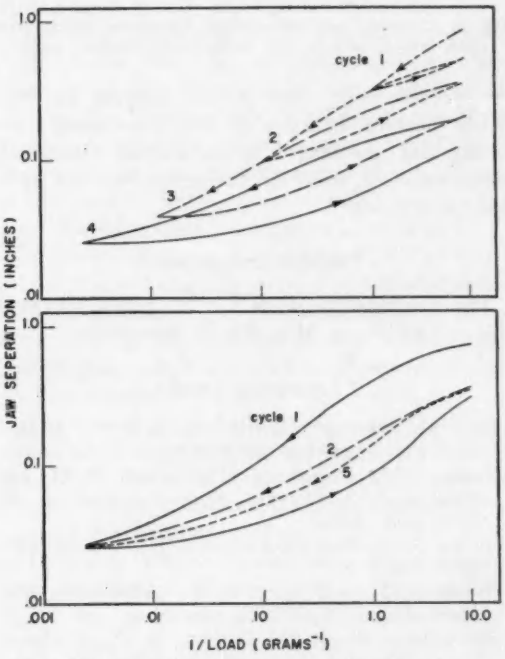


Fig. 5. Cyclic creasing load-deformation curves for cotton print cloth.

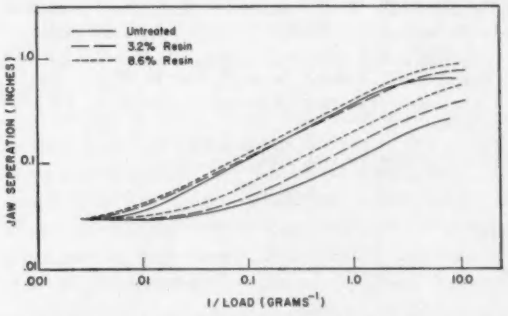


Fig. 6. Creasing load-deformation curves for cotton print cloth treated with a methylated urea-formaldehyde precondensate.

Effect of Crushproofing Resin

The confusion between terms like crease resistance and crease recovery in describing the properties of cellulosic fabrics treated with aminoplast resins indicates a lack of understanding of just what changes in fabric properties have been produced. The usual test methods, such as the T.B.L., Monsanto, or Shirley Institute procedures are measures of crease recovery and give no information on the resistance to creasing. Load-deformation curves obtained by the procedure given above should be informative in this respect.

In Figure 6 there are load-deformation curves for cotton print cloth before and after treatment with a methylated urea-formaldehyde precondensate. Within experimental error, the deformation curves appear to be the same, indicating that there has been no measurable change in the resistance of this fabric to creasing. The recovery curves and ultimate recoveries are different, however. The latter show reasonably good correlation with the crease recovery measured by the Shirley Institute procedure as shown in Figure 7, which includes data from two samples omitted from Figure 6 for the sake of clarity.

These results are in line with the conclusion of Cooke [2] that the ability of a fabric to recover from creasing is more important than its ability to resist creasing. However, Schiefer [6] found that an unspecified crease-resistant finish increased the work necessary to bend a number of fabrics in the flexometer. This could have been due to the use of a partially polymerized resin instead of a precondensate or to the addition of a hand-modifier in the resin treatment, and we have observed similar results with materials of this type.

Effect of a Surface Resin

When prepolymerized resins are deposited on fabrics, they stiffen the fabrics and change their hand

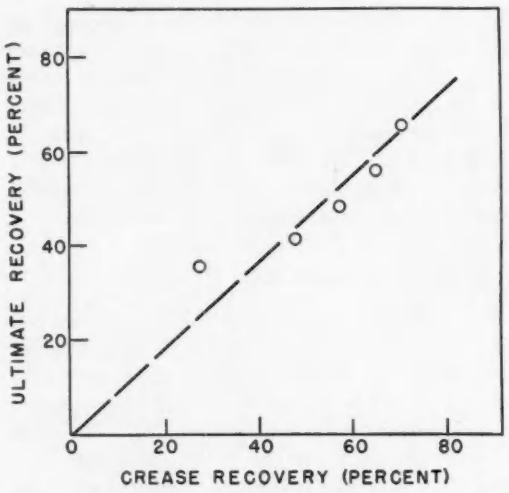


Fig. 7. Correlation between crease recovery and ultimate recovery from creasing deformation.

to a degree which depends on the mechanical properties of the resin itself. The effect of various concentrations of a relatively stiff resin of this sort on the creasing load-deformation curve of cotton print cloth is shown in Figure 8. The experimental resin used for this experiment was an acrylic copolymer whose second-order transition temperature is about 50° C. This means that at the temperature of the test it forms a relatively hard, stiff film which changes the properties of the fabric in this direction. The figure shows that as the concentration of the resin on the fabric increases, greater stiffness is observed in the initial part of the test, and the knee in the curve becomes more pronounced. This knee is probably the region where most of the fiber-to-fiber or yarn-to-yarn resin bridges formed within the fabric are broken.

Conclusions

By a relatively simple arrangement the Instron tester may be converted to a compression tester with which increasing load-deformation curves for fabrics

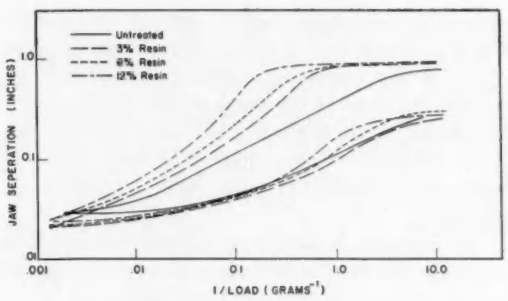


Fig. 8. Creasing load-deformation curves for cotton print cloth treated with a stiff thermoplastic surface resin.

can be obtained. Such curves describe the low-radius bending behavior of a fabric rather completely and are related to its stiffness, resilience, crease recovery, and other properties involved in its drape and handle.

Acknowledgment

The experimental work for this paper was very ably carried out by Mrs. Angela Simonson.

Literature Cited

1. Am. Soc. Testing Materials, Standard No. D1388–55T, Part 7, pp. 622–627 (1955).
2. Cooke, T. F., Dusenbury, J. H., Kienle, R. H., and Lineken, E. E., TEXTILE RESEARCH JOURNAL **24**, 1015–1036 (1954).
3. Finch, R. B., TEXTILE RESEARCH JOURNAL **18**, 165–177 (1948).
4. Hindman, H. and Burr, G. S., *Trans. Am. Soc. Mech. Engrs.* **71**, 789–796 (1949).
5. McNicholas, H. J. and Hedrick, A. F., *Technical Notes*, National Advisory Committee for Aeronautics, No. 335, "The Structure and Properties of Parachute Cloths," Washington (March 1930).
6. Schiefer, H. F., *J. Research Natl. Bur. Standards* **10**, 647–657, 705–713 (1933); *ibid.* **20**, 221–252 (1938); *Am. Dyestuff Reptr.* **26**, 667–669 (1937).
7. Zyrin, S. G., *Tekstil' Prom.* **5**, No. 10, 25–27 (1945); see also TEXTILE RESEARCH JOURNAL **19**, 239 (1949).

Manuscript received March 25, 1957.

Creslan[1, 2]

C. W. Bendigo

Fibers Division, American Cyanamid Company

FOR almost a half century, American Cyanamid Company has been serving the textile industry with its Calco dyes, textile resins, and textile specialties. Now it is embarking upon another facet of service to this industry with its Creslan acrylic fiber.

During World War II, Cyanamid became the country's—in fact, the world's—largest-scale producer of a new basic raw material, acrylonitrile. This material was needed for oil-resisting synthetic rubber essential for military uses. Textile fibers produced from it showed merit, and soon each acrylo market survey showed textile fiber uses as a good outlet for large quantities of acrylonitrile.

A similar situation occurred after World War I. At the end of hostilities there were available acetate facilities developed for the manufacture of acetate dope used to coat airplane fabric. It now seems natural that acetate fibers should have resulted from this war-developed material.

We do not often think of major textile developments as resulting from wars, but we have today at least three examples in the fiber field: acetate and acrylic fibers, as mentioned, and the widespread industrial and military use of nylon.

Following studies and work on a different type of fiber, research at Cyanamid on the production of fibers from polymers of acrylonitrile resulted in a new method of spinning acrylic fibers on which patents have been granted in the United States and many foreign countries. This Cyanamid-developed system involves fundamentally the spinning of a solution into a frigid bath, and is the basis for what we believe to be a versatile system for wet-spinning acrylic fiber.

Spinning a fiber from fiber-formable materials ordinarily is not particularly difficult. The problem is to spin a usable fiber, or, better still, a fiber with outstanding or unique properties. Because of the close association between Cyanamid's fiber development and its extensive textile dyestuff facilities, it is easy to understand why one of the most influential factors in the development of the acrylic fiber was to create a fiber which would be outstanding in dyeability. It was also found that this dyeability could be achieved while retaining other important properties.

The physical properties of Creslan, other than its superior dyeability in its field, are those which fulfill basic performance requirements in a wide range of consumer products. These include softness, high strength, light weight, wrinkle resistance, resilience, washability, dimensional stability, resistance to moths and mildew, flame resistance, and the ability to be durably creased or pleated.

Creslan processes well on equipment that has been found satisfactory for other acrylic fibers. Very even yarns generally result from staple Creslan acrylic fiber; the development in tow form is progressing.

In developing Creslan, our experience in the textile industry through our dyestuffs dictated that a fiber be produced which would dye inexpensively with good fastness when dyed in a wide range of colors and with several classes of dyes. Table I illustrates the dyeability of Creslan in 100% form.

It will be noted from this chart the large number of classes of dyes that are suitable. We are by no means saying that all dyes in all classes are satisfactory. That, as is well known, is not true for any fiber. We should also point out that although we have done our development work using, by and large, dyes that are currently used on the older fibers, we do not in every case get the same dyeing results. For example, a number of wool dyes have better fastness on Creslan than on wool.

Table II indicates the cost of dyeing 100% Creslan in light, medium, and dark shades. We feel that we have a particular advantage in lower cost for the dark shades—especially when fastness is considered.

For dyeing unions of Creslan and wool, we have found the acid, acid metalized, neutral metalized, chrome, and milling classes of dyes to be successful. The fastness of many of the dyes in the classes listed is limited by their fastness on the wool component.

[1] Trademark for American Cyanamid's acrylic fiber.

[2] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

TABLE I. Chart of Creslan Dyeability

Classes of dyes	Build-up	Fastness to: Light	Perspiration	Wash	Crocking
Disperse	Moderate–good	Moderate–good	Moderate	Moderate–good	Moderate–good
Basic	Moderate–good	Moderate–good	Moderate–good	Good	Good
Neutral milling	Good	Fair	Good	Good	Good
Neutral metalized	Good	Good	Good	Good	Good
Acid	Good	Good	Good	Good	Good
Acid metalized	Good	Good	Good	Good	Good
Chrome	Good	Good	Good	Good	Good
Direct	Good	Fair–good	Fair–good	Fair–good	Fair–good

TABLE II. Typical Costs of Dyeing 100% Creslan

Maize	Green	Brown
Disperse dyes Cost: 1.4¢/lb. Time: 2¼ hr.	Acid dyes Cost: 7.7¢/lb. Time: 3¾ hr.	Chrome dyes Cost: 6.7¢/lb. Time: 5 hr.

TABLE III. Typical Cost of Dyeing Creslan and Wool Unions

Rose	Red	Black
Acid metalized dyes Cost: 4.0¢/lb. Time: 2½ hr.	Milling dyes Cost: 8.8¢/lb. Time: 3 hr.	Chrome dyes Cost: 13.3¢/lb. Time: 4½ hr.

The dyes are employed in a one-bath method by procedures which ordinarily do not differ materially from those used in dyeing 100% wool.

Table III gives some indication of the cost of dyeing Creslan and wool unions. We believe the costs and the quality results obtained are outstanding.

American Cyanamid is in the process of building a plant near Pensacola, Florida. Until this plant begins production late in 1958, the only Creslan available is that produced in the prototype pilot plant at our Research Laboratories in Stamford, Connecticut. Thus we are able to work with only a small fraction of the concerns which have expressed an interest in this fiber.

Society of Rheology to Meet

The Society of Rheology will conduct its Annual Meeting at Textile Research Institute, Princeton, N. J., November 7, 8, and 9. Technical sessions will be held in the Edward T. Pickard Seminar Room.

Zefran[1]

G. W. Stanton[2]

Textile Fibers Department, Dow Chemical Co., Pittsburg, Cal.

THIS is the first presentation of the properties of Zefran, the new fiber scheduled for commercial production by The Dow Chemical Company early in 1958. The following is a description of the fundamental differences between Zefran and other synthetic fibers and will emphasize those properties which may be most useful to the textile industry.

Zefran is classified as a nitrile alloy fiber. This designation is considered to indicate correctly the fiber structure and to distinguish it from acrylonitrile copolymer fibers, which are usually referred to as acrylics. Before the nitrile alloy structure is described, brief reference will be made to the problem of fiber nomenclature. Polyacrylonitrile fibers are not as easily classified under a single word heading as the textile industry might wish. The difficulty can be visualized by comparison with the synthetic cellulose fibers. It was soon recognized that cellulose nitrate, cellulose acetate, and regenerate cellulose could not be lumped together under one designation. After the demise of fibers made from cellulose nitrate, the problem was simplified and has eventually resolved itself with the acceptance of two classifications, acetate and rayon. Although this takes care of most of the problems created by differences in chemical compositions, other problems are introduced by the spinning methods employed. Rayon produced by the viscose process is significantly enough different from that produced by the cuprammonium process to make different designations, such as viscose and Bemberg, desirable. With the recent introduction of cellulose triacetate fibers, the acetate picture is becoming complicated, and another major designation category is in the making.

The problems of classifying polyacrylonitrile fibers are equally complex. As shown in Table I, two major variables introduce significantly different sets of properties into the fibers derived from acrylonitrile; many of these differences can be covered by trade names. For example, whether the additive is acidic or basic makes little or no difference in the physical properties of the fiber, but imparts characteristic dyeability. Also, the differences in fiber cross section resulting from the spinning method may be adequately designated by trade names. The two variables which contribute the most important fiber property differences are (A) the percentage of modifier and (C) the manner of incorporating the modifier.

It is generally agreed that polyacrylonitrile fibers should contain over 85% acrylonitrile, which takes reasonably good care of (A). However, in order to classify properly according to (C), a distinction must be made between a copolymer and a blend, graft, or block polymer, we suggest the designations *acrylic* and *nitrile alloy* for this purpose.

Referring to Figure 1, an attempt is made to depict schematically the general difference between a copolymer structure and a blend, graft, or block structure. The rectangles indicate dye receptive groups; each polymer structure is shown to have the same number. These dye-binding groups are randomly dispersed in the copolymer, while they are joined together in the alloy, which can represent either a blend of polymers, a graft polymer, or a block polymer.

The high degree of hydrogen bonding between CN groups and hydrogens on adjacent chains of polyacrylonitrile is shown to be only slightly disturbed in the alloy structure. The dye-receptive groups in the copolymer structure are shown to disturb this uniform bonding tendency and open up the molecular spacing. Crystals of polyacrylonitrile are imperfect, at best, because of the random *d*- and *l*-

TABLE I. Types of Polyacrylonitrile Fibers

1. Differences in chemical composition
   - A. The percentage of modifier.
   - B. The kind of modifier; acidic, basic, nonionic, etc.
   - C. The manner of incorporating modifier; copolymer, blend, graft, block, etc.
2. Differences in spinning methods
   - A. Dry spinning.
   - B. Wet spinning from organic solvents.
   - C. Wet spinning from aqueous salt solutions.

[1] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

[2] Director of Research.

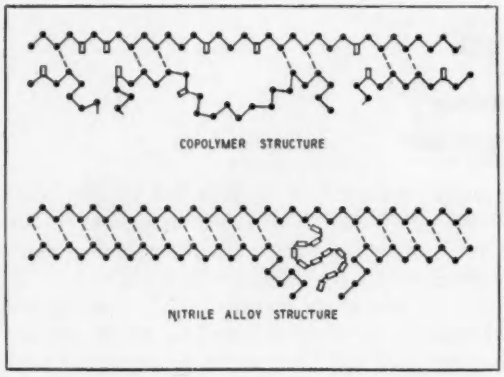


Fig. 1. A schematic comparison of the acrylonitrile copolymer structure with the nitrile alloy containing the same number of dye-receptive groups.

arrangements of CN groups along the chain and the exceptionally high concentration of hydrogen bonding points which it contains. However, it forms a highly impervious hydrophobic structure which is randomly loosed by most dye-receptive comonomers. The alloy structure is essentially a continuous hydrophobic polyacrylonitrile backbone, containing discrete volumes of a hydrophilic dye-receptive polymer. The close packing of the hydrophilic groups provides an atmosphere of the right cohesive energy density for tightly complexing dye molecules, which is not provided in a copolymer. This is particularly noticeable with vat, naphthol, and sulfur dyes. At the same time, the hydrophobic backbone remains intact to provide improved thermal properties.

The properties which most clearly distinguish the nitrile alloy structure from the acrylic copolymer structure are shown in Table II. We have prepared a copolymer having the identical composition of the Zefran alloy, as well as the same ultimate tensile properties of 4.4 g./den. and 30% elongation. The properties of the Zefran alloy and the acrylic copolymer are notably different in density and in crystalline structure. The moisture regain is higher for the alloy. The sticking temperature is about 35° F. higher for the alloy; this is related to safe ironing temperature and the tendency for hole melting, both of which are significantly higher for the alloy. One of the most pronounced differences shows up in dyeability. Both structures accept acetate dyes very well; however, the anthraquinone vat dyes show excellent build-up on the Zefran alloy and poor build-up on the acrylic copolymer.

TABLE II. Distinguishing Properties

	Zefran (alloy)	Copolymer (acrylic)
Density	1.19	1.17
Relative crystallinity (x-ray)	52%	44%
Average crystal size	122 Å	109 Å
Moisture regain		
65% RH	2.5%	1.6%
95% RH	4.7%	2.4%
Sticking temperature	490° F.	455° F.
Dye build-up		
Acetate	Good	Good
Vat (anthraquinone)	Excellent	Poor

Figure 2 further indicates the difference in thermal properties between the Zefran alloy fibers and the copolymer fibers of comparable composition. Both fibers were first heat-set under the same conditions (10 min. at 300° F.) and then subjected to boiling-water shrinkage. After 10 hr. the Zefran fiber has shrunk less than 2%, while the copolymer has shrunk over 12%. This is in spite of the fact that Zefran has a higher water absorption.

Figure 3 shows the range of tensile properties which Dow's process can impart to Zefran. The fiber could be made from as low as 1 g./den. and more than 60% elongation to over 8 g./den. and about 10% elongation. The center curve was chosen to give the best over-all fiber characteristics. The

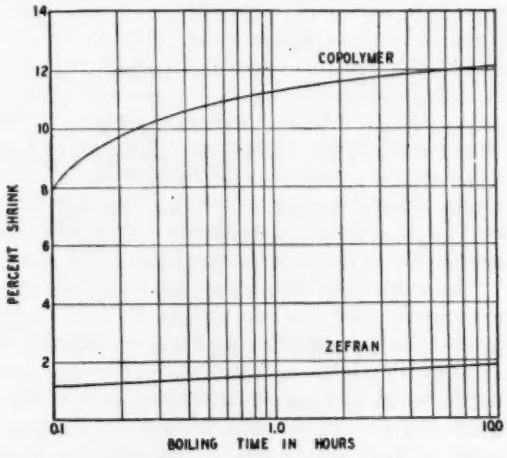


Fig. 2. Comparison of the shrinkage in boiling water of a copolymer fiber having the same ultimate tensile properties and composition as the nitrile alloy, Zefran. Both fibers were first heat set for 10 min. at 300° F.

area under the curve indicates that toughness, or ability to do work, is high. The abrasion resistance is good, yarn elasticity is high, etc.

In Figure 4 are compared the properties of the crimped and the uncrimped fiber. Properties indicated by the shape of the curves are, in general, desirable. The high initial modulus indicates crispness of hand, and the yield point is high for good shape retention; the ultimate properties of tenacity and extensibility are reflected in good durability. The crimped fiber curve indicates the properties of Zefran as it is normally sold. Crimp is essential for good processability on conventional textile equipment and imparts softness and resilience to the finished fabric. The crimp in Zefran is worth special mention in that it is permanent to boiling water. The crimp is put into the fiber early in its manufacture in such a way that the fiber can "remember" only its crimped condition. If the crimp is pulled out during processing, it can be recovered to a great extent by scouring or during dyeing.

Figure 5 compares the tensile curves for wet and dry Zefran fiber. The high modulus and yield point of the wet fiber show that it retains its crispness and dimensional stability while wet and does not become soggy or weak. The continuous hydrophobic crystalline backbone of polyacrylonitrile in the alloy structure permits it to retain 85–90% of its dry properties while wet.

At the same time is seen in Figure 6 the high moisture regain, which is contributed by the hydrophilic dye-receptive component of the alloy structure. The high moisture regain controls static to some extent, especially at higher humidities, and also imparts added comfort when worn next to the skin. The fiber is also quick wetting, and soils are removed easily by laundering.

Figure 7 shows the wrinkle recovery characteristics of Zefran fabric versus relative humidity. Here again the continuous hydrophobic backbone in the alloy structure contributes to excellent performance in this important property, required for wash-and-wear fabrics.

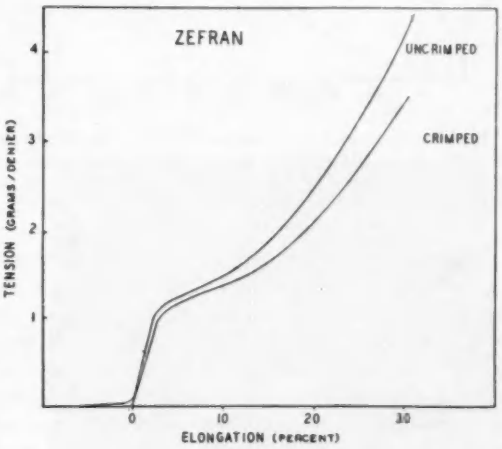


Fig. 4. Comparison of the tensile properties of Zefran with and without its normal crimp. The crimp is stable to boiling water.

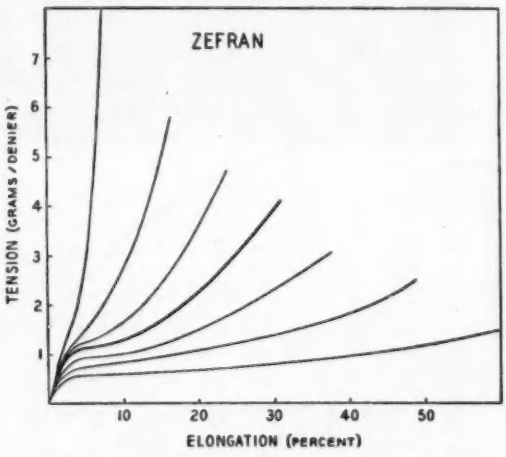


Fig. 3. The range of tensile properties which Dow's process can impart to Zefran fiber. The center curve is for the fiber regularly manufactured.

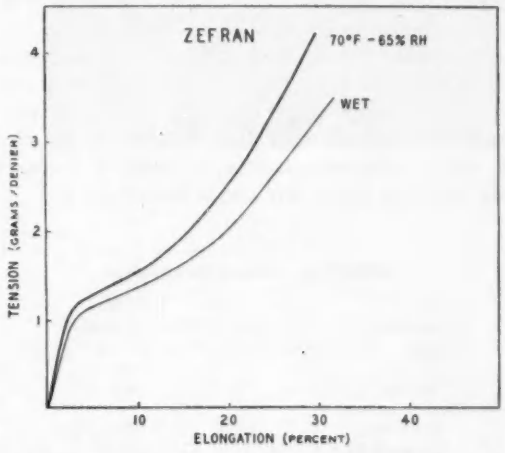


Fig. 5. Comparison of the wet and dry tensile properties of Zefran. Between 85 and 90% of the dry properties are retained when wet.

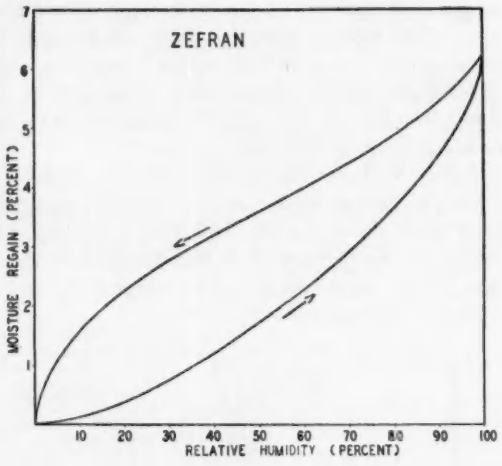


Fig. 6. Moisture regain curves for Zefran at 70° F. and 0–100% R.H. The upper curve is for equilibrium approached from the wet side, while the lower curve is obtained approached from the dry side.

Another property required for wash-and-wear fabrics, crease retention, is shown in Plate I. On the left is a 100% wool fabric and on the right a similarly constructed fabric of Zefran. The fabrics were creased and then subjected to a 120° F. washing with 0.5% soap for ½ hr. (AATCC No. 2). Not only does the Zefran completely retain its crease, but the smooth pressed appearance is retained in comparison to the mussed appearance of the wool fabric. Mussing is caused by swelling and shrinking of a fiber during wetting and drying; Zefran's water absorption is too low to cause this effect.

Additional physical properties of Zefran are shown in Table III. Table IV summarizes the chemical resistance of Zefran; all of the test results are satisfactory to excellent. Table V summarizes the effect of various durability factors; in addition, Zefran is not irritating to the skin and is non-allergenic.

TABLE III. Properties of Zefran

Color	White
Cross section	Round
Crimp	
%	24
No./in.	8.5
Tensile properties	
Tenacity	3.4 g./den.
Extensibility	30%
Modulus	37 g./den.
Yield point	1.05 g./den.
Shrinkage (boiling water)	1–2%

TABLE IV. Chemical Resistance of Zefran

Common solvents	Generally good
Acids (up to 40% conc.)	Excellent
Alkalis (at the boil)	Yellows
Mercerizing conditions	No effect
Detergents	Excellent
Fats and oils	Excellent
Dry cleaning solvents	Excellent
Odor retention	None

The dye receptivity of Zefran to the various classes of dyes is seen in Table VI. Satisfactory dyes are available for essentially all colors and all end uses. The neutral premetalized acids and after treated directs are particularly recommended for beck work and knit goods. The after treated directs, naphthols, sulfurs, and vats offer especially good opportunities in blends with cotton and rayon, or with 100% Zefran in the flat width and continuous dyeing methods. Note particularly the anthraquinone vat dyeability of Zefran. This class of dyes is recognized to have outstanding fastness properties to both sunlight exposure and high-temperature laundering. Its use has been fairly well limited to cotton and rayon applications; most man-made fibers have shown no affinity for it when applied at temperatures under the boil.

It is impossible to cover the complex field of dyeing, with its many methods of dye application and problems encountered in dyeing blends with other fibers. However, this article includes colored il-

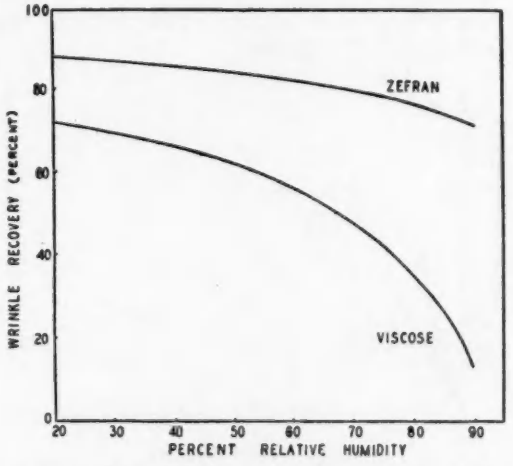


Fig. 7. Comparison of the wrinkle recovery properties of Zefran fabric and viscose rayon fabric of the same construction at 70° F. and varying relative humidity.

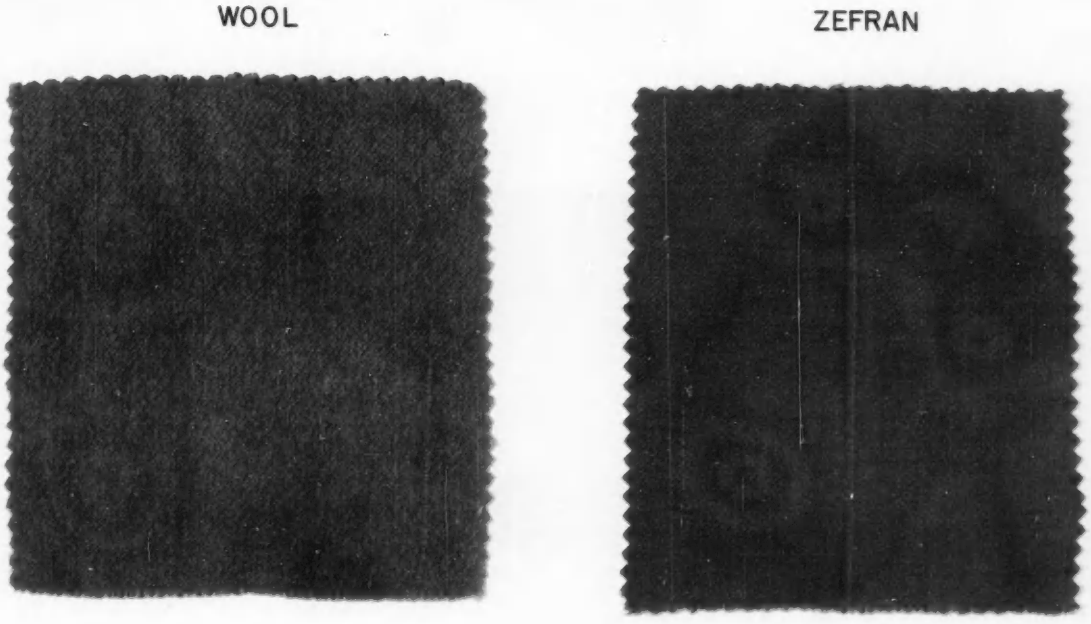


Plate I. Comparison of the crease retention of Zefran fabric (right) with a similarly constructed wool fabric (left) after wash testing at 120° F. (AATCC No. 2). Note the complete retention of crease and the absence of mussing in the Zefran fabric.

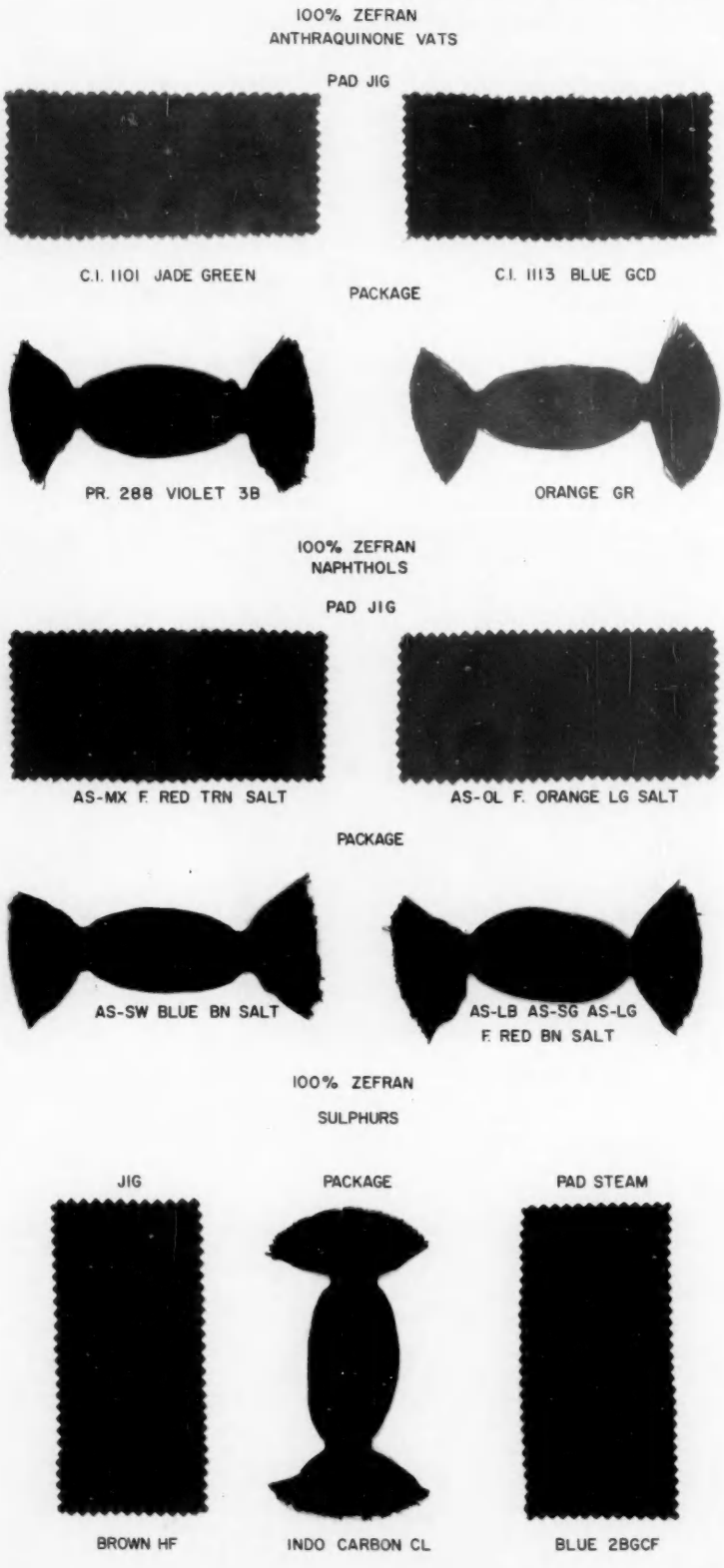


Plate II. Anthraquinone vat dyes applied to Zefran fabric and yarn using conventional cotton dyeing procedures and temperatures in the range of 100–180° F.

Plate III. Naphthol dyes applied to Zefran fabric and yarn. Coupling of the naphthol and salt is started at about 65° F. and raised to 100° F. for 15–20 min.

Plate IV. Sulfur dyes applied to Zefran fabric and yarn. Note the dark shades obtainable with this inexpensive class of dyes.

ZEFRAN-COTTON BLEND (50-50)

Plate V. Union dyeings of Zefran and cotton blends after mercerization. Excellent unions are obtained with anthraquinone vats and naphthols using conventional procedures.

100% ZEFRAN PAD STEAM

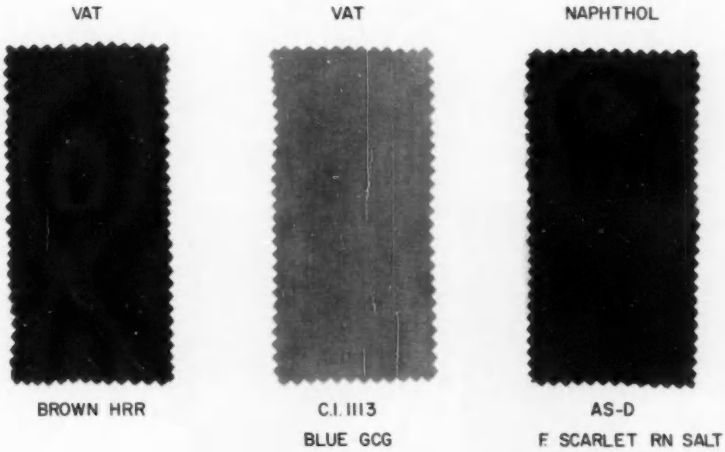


Plate VI. Zefran fabrics which have been continuously dyed by the pad steam process using anthraquinone vats and naphthols. This represents a high-speed, cost-saving operation for long yardages. Steaming times were 20 sec.

100% ZEFRAN PRINTS

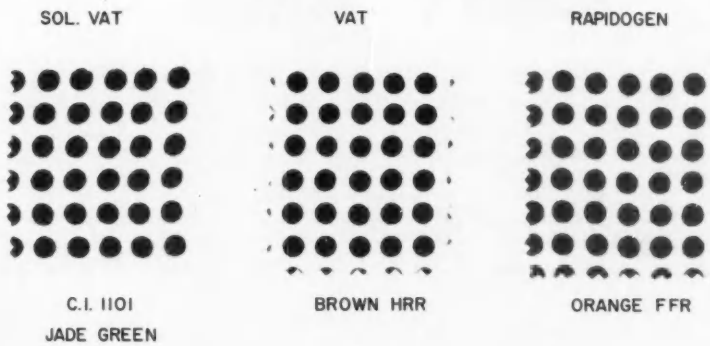
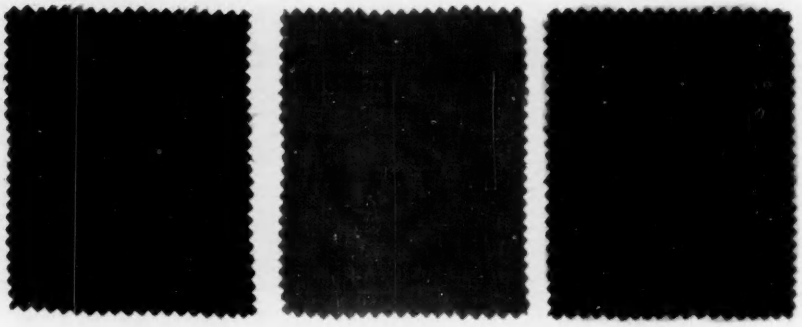


Plate VII. Zefran fabrics printed by conventional techniques using soluble vats, anthraquinone vats, and rapidogens. Both machine and screen printing are applicable.

ZEFRAN-RAYON BLEND (50-50) BECK DYED

AFTERTREATED DIRECTS



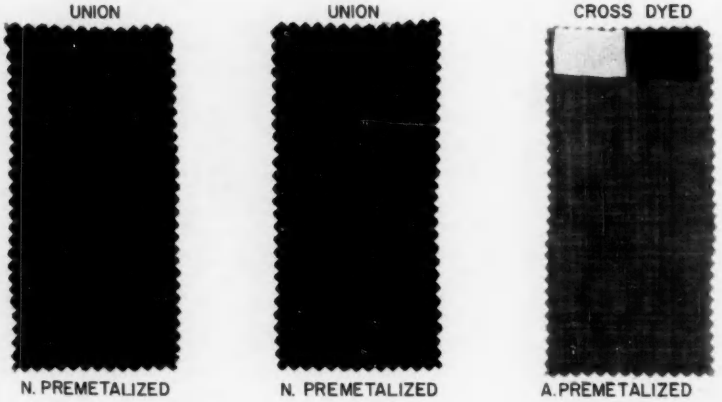
FAST BROWN PR.47 | RESERVE CROSS DYE | NAVY BLUE SL

Plate VIII. Union and cross dyeings of Zefran and viscose rayon using after treated direct dyes. Excellent unions in a wide range of shades are obtained using single bath methods. The cross dyeing is obtained using a special reserving agent for Zefran.

ZEFRAN WOOL BLEND (50-50)

BECK DYED

UNION | UNION | RESERVED CROSS DYED



N. PREMETALIZED BROWN RL YELLOW 2RL | N. PREMETALIZED BLACK NB | A. PREMETALIZED BLUE FR YELLOW N RED GL

Plate IX. Union and cross dyeings of Zefran and wool. Excellent unions are obtained with neutral premetalized acid dyes using short cycle single bath procedures conventional for wool. Heathers are produced with acid premetalized dyes by employing a reserving agent for the Zefran.

TABLE V. Durability of Zefran

Weathering	Excellent
Insects and microorganisms	No attack
Abrasion resistance	Very good
Pilling resistance	Excellent
Hole melting	Equal to wool
Launderability	Excellent
Appearance retention	Superior

TABLE VI. Dyeability of Zefran

Class of dye	Color build-up	Fastness		
		Light	120° F. wash	160° F. wash
Acetate	Good	Poor	Poor	Poor
Acid	Fair	Fair	Fair	Poor
Acid premetalized	Fair	Good	Good	Fair
Neutral premetalized (R)	Good	Good	Good	Fair
Basic	Fair	Poor	Poor	Poor
Direct	Good	Fair	Fair	Poor
Direct after treated (R)	Good	Good	Ex.	Good
Naphthol (R)	Good	Ex.	Ex.	Good
Sulfur (R)	Good	Good	Ex.	Good
Soluble vat (R)	Good	Ex.	Ex.	Good
Vat (indigoid) (R)	Good	Good	Ex.	Good
Vat (anthraquinone) (R)	Good	Ex.	Ex.	Ex.

R = Recommended.

lustrations in order to give an over-all impression of Zefran dyeing.

Plate II shows the application of anthraquinone vat dyes to 100% Zefran by both pad jig and yarn package dyeing. Temperatures employed here were in the range of 100–180° F. Notice the depth of shade and the cleanness of color.

Plate III shows the application of naphthol dyes to 100% Zefran by both pad jig and yarn package dyeing. Conventional procedures were employed throughout. Here again, notice the dark shades obtainable and the brilliance of color available with this class of dyes.

Plate IV shows the application of sulfur dyes to 100% Zefran. These dyes are particularly inexpensive and produce excellent wash fastness in heavy shades.

Plate V shows the union dyeing of a blend of Zefran and mercerized cotton. The fabric was dyed by the pad jig procedure (one vat and one naphthol). The yarn sample was package dyed with a vat. Excellent unions are obtained with both vats and naphthols.

Plate VI shows 100% Zefran fabric continuous dyed with vats and naphthols. The procedure used here was continuous pad steam, and the steaming times were 20 sec. The Williams unit may also be used. This labor-saving method of dyeing long yardages of fabric offers significant cost savings for many applications and is another advantage of Zefran over many man-made fibers which cannot be handled by continuous dyeing methods.

Plate VII shows the printability of 100% Zefran fabric with vats, soluble vats, and rapidogens. Both machine printing and screen printing methods are applicable; conventional cotton procedures are followed.

Plate VIII shows the union dyeing of Zefran and rayon using after treated direct dyes. The beck dyeing procedure was used here. Good unions having good fastness properties can be obtained in all depths of shades. When cross dyed effects are desired, as shown by the center fabric, a special Zefran reserving agent is available.

Plate IX shows the union dyeing of Zefran and wool; excellent results are obtained with simple, conventional procedures. These fabrics were beck dyed using neutral premetalized dyes for the unions and acid premetalized dyes for the heather or cross dyed effect. Single bath methods and relatively short dyeing cycles commonly used for wool can be used with Zefran–wool blends to obtain a wide range of shades with good fastness properties.

The forms of Zefran which are currently available are shown in Table VII; other deniers and staple lengths will be made available as uses are developed for them. Zefran has been handled on all conventional textile equipment with very good results. The sharp, durable crimp in the fiber, combined with selected processing finishes, permits high volume output on most equipment with good product uniformity.

Loft, or high bulk, is one of the notable properties of Zefran. This property can be enhanced by combining staple fibers having different shrinkages or by stretching and combining tows on the Turbo stapler or the Pacific Converter. These procedures offer no unusual problems.

Over one hundred different potential end uses have been investigated on a development scale, using pilot plant produced Zefran fiber. Many of these

TABLE VII. Available Forms of Zefran

Deniers	2 and 3 den./f.
Staple lengths	1½, 2, 2½, 3, 4½ in.; tow
Lusters	Bright and semi-dull
Dyeabilities	Regular and nonstaining

appear commercially promising and will be in production when Zefran fiber is available in quantity at the beginning of next year. Men's, women's, and children's apparel—in both knitted and woven fabrics from 100% Zefran and in blends with cotton, rayon, and wool—appear to be excellent applications for the fiber.

Extensive wear tests have been conducted on a wide variety of garments. The outstanding characteristic noted for Zefran is the excellent appearance retention during the long life of the garment. Other markets, such as household goods—including blankets, draperies, and upholstery—are found to benefit by the properties which Zefran can impart. In the textile areas where the fastness properties of vat dyes are required in combination with wrinkle recovery, crease retention, low shrinkage, and general ease of care, Zefran has much to offer.

Cotton Merchandising Changes to Conform with Modern Mill Requirements[1]

Earl E. Berkley

ACCO Fiber & Spinning Laboratory, Houston, Texas

Historical Background

Prior to World War I, simple gin plants without driers and cleaners were common. They were widely used until mechanical harvesting was accepted, since they were adequate for ginning carefully hand-picked cotton which had little or no trash. They were also built to gin cotton that had been dried either in the sun or in a cotton house.

During World War I, hand picking gave way to hand snapping; in 1914 the mechanical stripper appeared in certain fields on the high plains of Texas.[2] Crude mechanical strippers were developed during the war for short staple cottons, but they were limited in number and type until about 1926. They are now a major factor in production, even in central and south Texas. The spindle picker, which was first tried out in 1926, is now a major economic factor, particularly in Arizona and California, as well as the central belt.

The shortage of labor in 1926 also resulted in an increase in the number of hours per day the mechanical harvesters were operated, and necessitated picking cotton under unfavorable conditions, such as in the early morning when the dew is on and shortly after showers. This made it necessary to put driers in the gins for removing this excess moisture.

When the spindle picker became established, there was so much trash brought in with the cotton that it soon became necessary to develop seed cotton cleaners. They were in general use at the beginning of World War II. These machines removed as much trash as possible before the lint was ginned from the seed. More recently, it has been necessary to develop stick machines to remove the branches of the plants which are broken off by the strippers.

The armed forces in World War II required the greatest production of materials of all similar activities in the history of mankind. Their manufacture utilized both skilled and unskilled labor *en masse*. Wages were good in the war industries, and many of the cotton tenant farmers and the farm owners themselves moved to the factories. This movement stimulated the mechanization of essentially all farm activities, except perhaps on the small farms. Cotton production, including harvesting, ginning, and warehousing, was mechanized. Automation in cotton manufacturing is now in full swing, partly as a result of labor shortages.

The gin stands themselves have been made stronger and more perfect in action, and now in-

[1] Presented at the annual meeting of the Textile Research Institute, New York City, March 15, 1957.

[2] Data on the origin of harvesting machinery were furnished by Fred C. Elliott, of A & M College, College Station, Texas.

clude mote knives for removing the motes, one of the most troublesome contaminants of raw cotton. Following the ginning, lint cleaners have been inserted in the lint flues. The lint cleaners have been in the process of development for many years, but were more or less generally accepted in 1947. The lint cleaners are of two major types: (1) the saw type cleaner; and (2) the air separator type.

The saw type cleaner has a lickerin type saw, similar to a card, which has bars or slots at a fixed distance from the saws over which the cotton is dragged (Figure 1). The trash is shaken free of the lint and allowed to drop through the bars; the lint is carried on by the saws and is doffed after passing the bars. These lint cleaners remove sufficient trash to raise the grade $\frac{1}{2}$–2 grades in extreme cases.

In the air flow type of lint cleaner, the cotton is taken from the gin into an air blast, which carries it across a slot at an elbow in the conveyor pipe (Figure 2). The cotton and trash strike the slot at high speed; a vacuum in the pipe beyond the slot draws enough air in through the slot to prevent the cotton fiber from passing out. The cotton is drawn back into the system, and heavy trash, having a greater density, is expelled into the trash chamber and trapped. The air flow cleaner does not remove all of the light leaf trash, which shows up as pin trash. The classer's grades are increased, but frequently not as much by the air flow cleaner as is possible when using the saw type cleaners. The air flow cleaners do, however, remove sand, dust, and trash, particularly motes and other heavy materials. The air flow cleaners frequently produce a sample with less mill waste than corresponding samples from the same lot of cotton cleaned on the saw type cleaners, although the latter may be slightly higher in classer's grade. The air flow cleaners also reduce the number of trash particles in the yarn and often result in economies to the cotton mills, but not to the farmer who is dependent upon the farm loan for his price on a portion of his cotton.

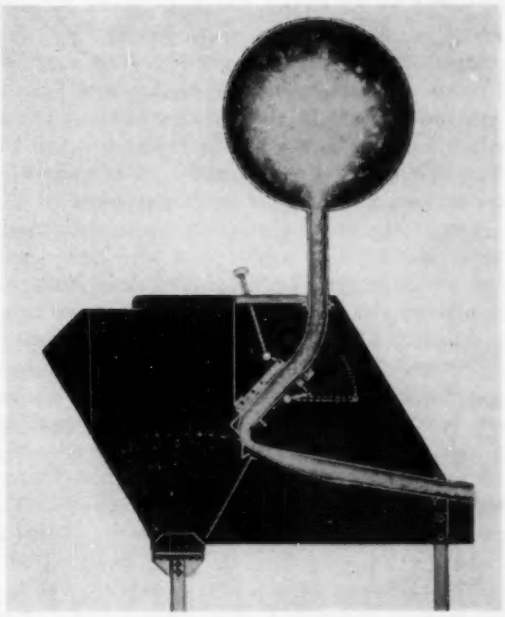


Fig. 2. Cross section of air flow type lint cleaner (courtesy Lummus Cotton Gin Co.).

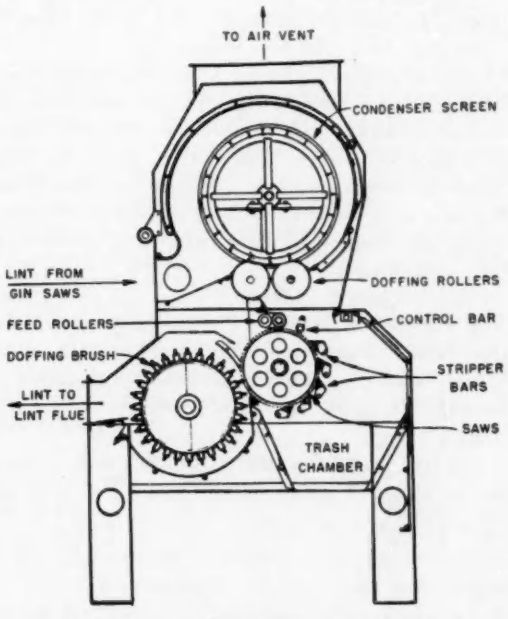


Fig. 1. Cross section of saw type lint cleaner (courtesy Continental Gin Co.).

Along with the changes in gin equipment, it has been necessary to change the presses also. The old gin press produced the low density, or flat bale, with a density of about 12 lbs./cu. ft. A 500 lb. bale occupies about 42 cu. ft. Cotton from Arizona and California, where practically no cotton was grown before World War II but where a major portion of the cotton is now grown, must be pressed to standard density or high density in order to conserve space

in transit to the mills. Standard density bales are pressed to about 26 lbs./cu. ft. (about 19 cu. ft.), whereas high density bales are 32 lbs./cu. ft. (about 15.6 cu. ft.). The gins in West Texas and farther west now have, for the most part, gin density presses which make a bale of about 26 lbs./cu. ft. This bale is completely covered; in many cases, even when the samples are taken, the patch is replaced.

One of the greatest needs in the cotton industry today is the establishment of a semi-high density bale which is pressed at the gins to about 30 lbs./cu. ft. and completely covered. Bales of this type would occupy about 17 cu. ft. They would pack closely in a ship, and, because of their uniform size and shape, would take up less space than our present high density bale. They would also be cheaper to warehouse in the United States. It is possible that the warehousemen could afford to abandon the now wasteful and expensive practice of repressing the bales. This is, in my opinion, an absolute must for American cotton.

The movement of industry, particularly the textile industry, into the southeastern states has created new uses for the land in those areas; now the farmers are growing dairy and poultry products, meats, vegetables, small fruits, etc. for the mill worker. Mechanized cotton production is better adapted to the larger farms prevalent in the Mississippi Valley, West Texas, and the irrigated states of New Mexico, Arizona, and California. The Southeast is destined to lose most of its cotton production to areas farther from the markets, partly because cotton is not perishable and can be stored indefinitely or transported long distances inexpensively by freight.

Cotton Quality and Properties

Texas now grows approximately one third of the total cotton produced in the United States; California is the third state in volume of production, and Arizona claims the honors for the highest yield per acre. Before World War II, a yield of 2 bales/acre was considered an excellent crop. Today, the Arizona farmers average throughout the state about 2¼ bales/acre. It is unofficially reported that one farmer this past year averaged 4 bales/acre on a 2200 acre planting.

In general, cotton grown in the arid regions is more uniform and is freer from defects such as field damage than cottons grown in the more humid central and eastern climates. In the irrigated region, when water is available the cotton can be kept fruiting right up to frost time. This is particularly true in the high plains area of Texas, where the growing season is relatively short. Large quantities of immature cotton are produced in certain seasons, and a limited amount every season on the high plains of Texas and farther west. Cotton fibers contain considerable sugar and proteinaceous materials in the protoplasm; these residues will cause the cotton to be spotted, tinged, or yellow-stained. This organic material tends to turn yellow, and even a light buff or brown color will develop in the cotton over a period of several months. Cotton of the same degree of maturity, however, that is defoliated by frost or chemicals, but not killed, will use up most of the organic materials before the bolls open, so that the cotton is white. It will remain white more or less indefinitely if it has been allowed to stay on the plant long enough to consume all reserves. If it is dried rather rapidly and the bolls open before these reserves are used up, color will develop, but it will develop more slowly and will be of lower intensity than in the bolls killed outright by freeze. These immature cottons will produce excellent fabrics in spite of the color if the cell walls have developed to a point where the Micronaire readings are 3.5 or above, but neppy yarns will result if the fibers are finer than 3.0 on the Micronaire.

In the irrigated regions, where the alkaline content of the soil is high, there is some indication that the alkali reacts with the wax on the cotton fiber, which in turn gives difficulty in processing, particularly in wet twisting. A waxy substance, that appears to be a soap-like material such as would result upon reaction of alkali with wax, accumulates on machine parts.

When cotton is defoliated too early, new growth will start before picking. The spindle picker crushes the tender leaves and causes chlorophyll stains. These stains have been somewhat of a problem, although the mills have now learned to cope with them.

For the past five years, ACCO has made a study of the effects of ginning procedures on the fiber and spinning quality of cottons. These studies have included the influence of speed of gin saws, the effects of moisture content of the cotton, and the effects of lint cleaners. They are being continued; the following preliminary data are of interest.

TABLE I. Grade and Staple Lengths of Cottons Ginned Dry (3% Moisture) and Humidified (6.5% Moisture)

Sample no.	Classer's grade		Staple length, in.	
	Dry	Humidified	Dry	Humidified
1	SM Lt. Sp.	SM Sp.	$1\frac{1}{16}$	$1\frac{1}{8}$
2	SM Sp.	SM Sp.	$1\frac{1}{8}$	$1\frac{5}{32}$
3	SM Lt. Sp.	SM Lt. Sp.	$1\frac{1}{8}$	$1\frac{1}{8}$
4	GM Sp.	SM Sp.	$1\frac{1}{32}$	$1\frac{1}{8}$
5	GM Sp.	SM Sp.	$1\frac{1}{32}$	$1\frac{1}{16}$
6	SM Lt. Sp.	SM Sp.	$1\frac{1}{32}$	$1\frac{1}{8}$
7	SM Sp.	SM Sp.	$1\frac{1}{32}$	$1\frac{1}{8}$
Mean	SM Sp.+	SM Sp.	$1\frac{1}{16}$	$1\frac{1}{8}$

In the irrigated regions, particularly during a dry season, cotton is dried to about 3% moisture in the field. If hand harvested and ginned as it comes from the field, the fibers may be broken to the extent of losing from $\frac{1}{32}$ to $\frac{1}{16}$ in. in staple length (Table I). Fiber arrays and spinning tests confirmed these losses in fiber lengths.

In the lint cleaner studies, it has been found that the air flow lint cleaners remove the heavy trash, thus reducing the mill waste; little of the spinnable fiber is lost. These cleaners remove a large percentage of the motes also, leaving a sample that can be processed into a yarn with a higher yarn grade than samples of the same cotton taken ahead of the lint cleaners.

The saw type cleaners remove a high percentage of the leaf trash, thereby increasing the classer's grade; they also remove a portion of the spinnable fiber. The saw type cleaners decrease the fiber length uniformity, which in turn is reflected in the yarn properties. The yarn appearance (Figure 3) and the yarn skein strengths decrease (Figure 4) as the classer's grades increase. These decreases are highly significant; the mill pays for a better cotton as the grade increases, but actually receives a poorer one. The advantages of the saw type cleaner are confined to the farmer, therefore; improvement of classer's grade brings a considerable premium in most seasons.

Fiber fineness, as measured on the Micronaire, is a major problem in cotton processing. Fineness is defined as the linear density, and may be expressed in weight per inch or as Micronaire readings. The Micronaire readings are influenced by fiber diameter (Figure 5) or intrinsic fineness, and by cell wall thickness (Figure 6) or maturity fineness. When using an American Upland cotton with a given staple length, for example, Micronaire readings are associated with differences in cell wall thickness. The old terminology of maturity is misleading, because cottons grown under drought conditions or on extremely poor soils may have thin-walled fibers when fully mature, whereas cottons killed by frost or freeze may have relatively thick fiber walls and still be immature. The immaturity would be indicated by the color and the percentage of water extractables.

Cotton maturity fineness has been associated with yarn appearance grades. The yarn appearance grades increase with an increase in Micronaire readings (Figure 7). For a given yarn number, the number of ends down increases as the Micronaire readings increase; other things being equal, the yarn strengths decrease. The gain in yarn appearance is

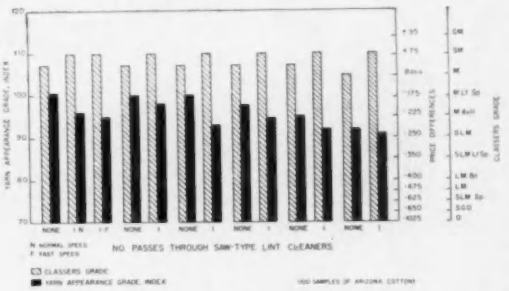


Fig. 3. Yarn appearance grades and classer's grades before and after saw type lint cleaners. (Joint program with Lummus Cotton Gin Co. and Institute of Textile Technology.)

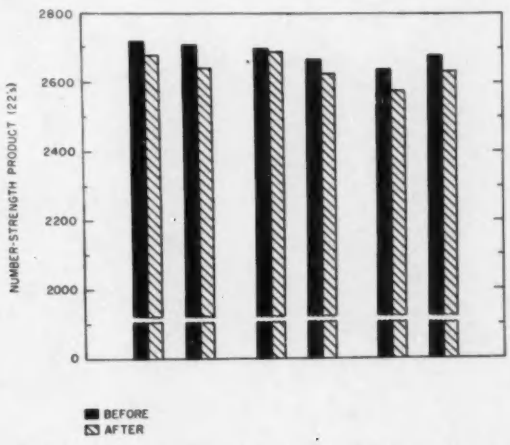


Fig. 4. Yarn strengths (break factor) before and after the saw type lint cleaner. (Joint program with Lummus Cotton Gin Co. and Institute of Textile Technology.)

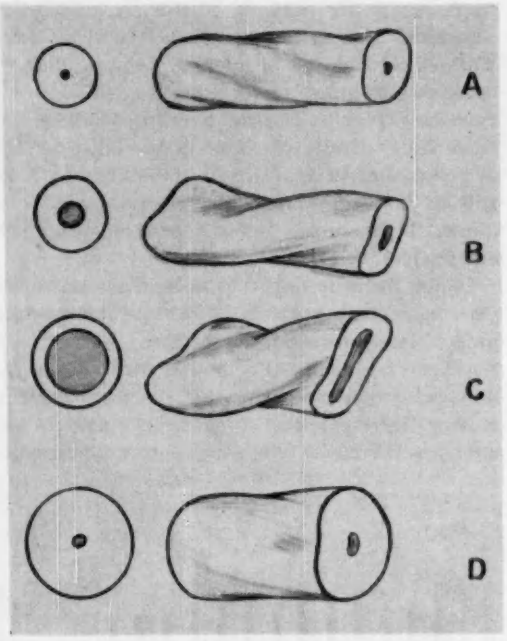


Fig. 5. Diagrams of diameters of types of cotton fibers found in world commerce: (a) Triple Hybrid, (b) Sea Island, (c) American Upland, and (d) Asiatic.

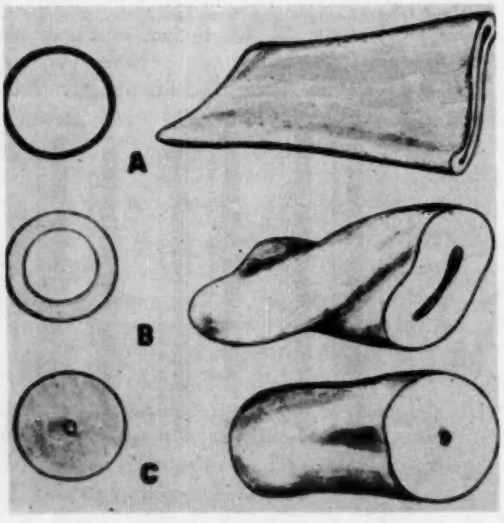


Fig. 6. Diagram showing variations in cell wall thickness: (a) thin-walled immature fibers, (b) medium thick fibers, and (c) thick-walled fibers.

offset by a loss in yarn strength and a greater cost in spinning. Fine fibers are more difficult to clean and produce more waste (Figure 8) than coarse cottons of the same grade and staple, but they require less twist in spinning. It is necessary, therefore, to balance cost of operation and yarn and fabric quality when choosing raw cottons for specific fabrics.

Maturity fineness also influences take-up in the loom; the finer the fiber, the greater the take-up (Figure 9). Fiber fineness is also associated with dyeing and finishing. Skein strength, loftiness, and other fabric qualities may be varied considerably by varying the fineness. Any given twist in roving and spinning is more effective for fine than for coarse fibers in the same constructions.

Defects in Cotton

Color in cotton is of considerable interest at this time because of the Government Loan. Certain cottons stored in warm moist climates become yellow to buff after a few years. This color is confused with the light tan to brownish red color associated with immaturity caused by a freeze.

There are at least five sources of color: (1) aging in storage, (2) immaturity from a freeze or other means that kills the fibers suddenly, (3) dirt or soil, such as tar, (4) minerals, and (5) field damage associated with microbiological decay. There may be any one, several, or all of these represented in a sample of low grade cotton.

All cottons tend to turn yellow with age, particularly if stored in a warm moist place. Bleached fabrics in the form of garments also tend to yellow. Cottons yellow at different rates, depending upon the origin. It is not entirely clear why certain cottons change faster than others, but the presence of sugars and proteinaceous materials in the residue of the protoplasm may be a factor. These organic substances appear to be a major factor in the color of immature cottons.

Asphalt tar is no doubt the most troublesome factor in soiling of raw cotton. Dirt, oil, grease, and other contaminants can be removed more easily than tar. In certain areas, red soil or clay may stain the fiber; red clay is also difficult to remove. The most difficult color to remove is that caused by the fungal spores associated with field damage. Certain fungi grow on the cotton; when they sporulate, the cotton turns gray or other shades, depending upon the

species. These spores are difficult to bleach white; when they occur in the lumen of the fiber, they cannot be washed out. The fungal mycelia also contribute color, which may be yellow, red, amber, brown, or black.

In addition to the color associated with these fungi, certain forms seem to cause a fluorescence; the fungus *Aspergillus flavus* is a good example. This fungus enters the boll before it is open through a worm puncture, or by other means, and feeds on the organic matter in the fiber. The fiber is weakened by the growth and may be considered perished. The small tufts of fibers containing this fungus glow under ultraviolet light with an olive-green fluorescence and are referred to as cat eyes. The fluorescence washes out in water, and there appears to be no deleterious effect on the bleaching, but the strength may be impaired. There are no reliable data as yet on the effects of this cat eye on dyeing and finishing.

The presence of cat eye has called attention to the fluorescence of cottons in general. Mills use ultraviolet light to separate samples of raw cotton and yarns. They have associated the appearance of the cottons with the dyeability of the yarn and fabric. There are several forms of fluorescence, and it is not clear as yet which of these are really responsible for the differences in dyeing.

Immature cottons yellow faster than mature cottons; after taking on a creamy or brassy cast, they often glow under ultraviolet light with varying intensities from one spot to another. Some spots look almost white, and others a bright yellow. A portion of the sample may have the bright halo with a slight purple cast characteristic of high grade bright cotton. The yellow and whitish casts under ultraviolet light appear to be associated with microorganisms feeding on the organic matter in the cotton. If so, this type of fluorescence could be associated with prematurely opened cottons.

Oil or grease shows up as a blue spot or cast under ultraviolet light. Chlorophyll stains have a rust color, and dusty or overheated cotton may have a deadpan appearance. It is not possible as yet to use the ultraviolet light to segregate cottons on the basis of overheating.

It is indicated that the most significant fluores-

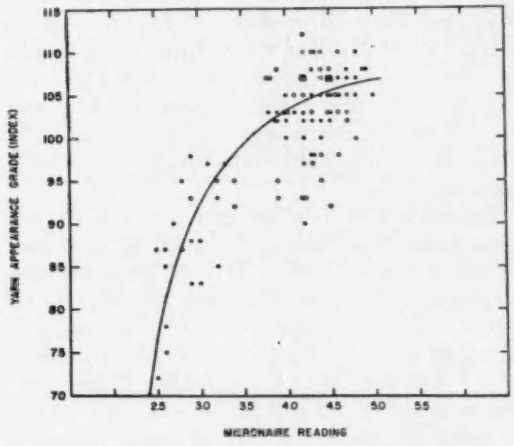


Fig. 7. Micronaire readings and yarn appearance grades.

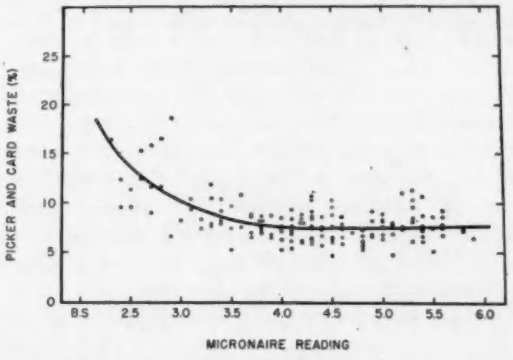


Fig. 8. Fiber fineness and percentage of waste in cottons of the same grade.

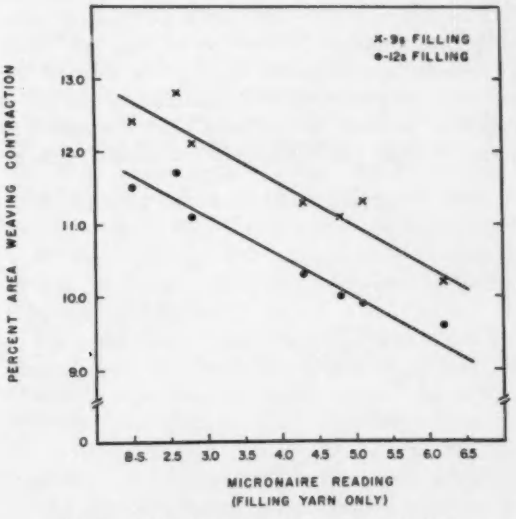


Fig. 9. Fiber fineness and take-up in the loom.

cence observed under the ultraviolet light is that associated with the presence of unusually high percentages of minerals such as iron. Cottons of that type have a lead cast under the ultraviolet light. Our studies are not adequate as yet to differentiate the samples on the basis of one element, but those known to have high percentages of iron have been observed to be different from other samples. These differences are more easily observed in the yarn and fabric than in raw cotton.

The effects of minerals in cotton are not known as yet, but they may account for differences in bleaching and dyeing that have been observed in cottons of the same fineness and in samples from different areas, such as irrigated versus raingrown cottons. The presence of iron in excess of 75 parts per million is associated with poor bleaching; quantities below 25 parts per million are associated with unusually high whites in bleaching. The high iron content can be recognized by a pinkish-yellow cast in the bleached yarn or fabric. It can be removed by the use of sequestering agents that tie up the iron, but this is expensive.

Minerals appear to have an effect upon dyeing. This may be due to the pH, since samples from high alkali areas in the irrigated region contain enough minerals to make soft wash water hard. The amount of each element and its hardness effect are not known as yet, but the problem appears to warrant careful study.

These and many other factors in a raw material such as cotton, which comes from all types of soils, complicate a modern industry more than they did a less precise one in earlier days. The demand for precision in processing and finishing of cotton is such that research on problems of this nature is a must. Simple problems, such as mistaking mote fragments in the yarn for pin trash, have led to the installation of millions of dollars worth of cleaning equipment to remove trash from high grades. Unfortunately, the excess beating given the cotton made the defects more prevalent. The motes broke up in processing, particularly in the card. The black fragments have fibers attached to them; these fibers spin into the yarn, and the black specks appear as pin trash. They do not shake off easily in winding, and have been observed to account for 75% of the trash defects in yarn made from high grade cottons. Strict Middling and better cottons may produce better yarns if de-moted on an air flow type cleaner and passed directly to the picker rather than processed on conventional mill equipment. The problem of cleaning cotton will bear a lot of study. Samples ginned on simple saw gins that produce grades as low as Ordinary to Strict Good Ordinary have been found to produce better yarns than the average Strict Middling cottons from the same general area of growth ginned on a modern gin with burr machines, seed cotton cleaners, and lint cleaners. When the cotton is overworked, either in the gin or in the mill, poor results can be anticipated in spinning.

Breakdowns in mills are now traced to the real cause rather than blaming the so-called "character" or lack of it in the cotton. In 1949, most difficulties encountered in cotton mills were attributed to the cotton, without attempting to see what might be wrong in the mill. Today only a small percentage of the difficulties are unrecognizable; if the cotton quality is known, the mill can, as a rule, be adjusted to make the desired quality of yarn. However, there are still many unsolved problems, and there will be as long as we continue to improve the old fabrics and construct new and better ones.

Orlon[1] Acrylic Fiber[2]

W. W. Heckert

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THE Orlon of today is an acrylic fiber with excellent whiteness, made from polyacrylonitrile, slightly modified to give it high affinity for basic dyes. It is the latest result of a continuing search for ways and means to build versatility into Du Pont's acrylic staple.

Its predecessors included an experimental staple modified to accept acid colors and a commercial variety which could be dyed by the cuprous ion technique. Experience with both of these versions soon made it evident that greater versatility and dependability were needed to enable the textile industry to take full advantage of the functional and aesthetic values latent in polyacrylonitrile. Careful analysis indicated that versatility could best be attained by simultaneously building into the fiber superior initial whiteness and dyeability with a class of dyes not ordinarily used on other fibers.

Type 42 Orlon, introduced in 1953, was the first acrylic fiber to embody this combination of properties. It had affinity for basic dyes and the best initial whiteness seen in an acrylic fiber up to that time. Since then some seventeen million dollars have been spent to improve and to service Orlon. New forms of the fiber and new technology for handling it have been introduced each year. Since this research effort has given the textile industry a number of new tools to use in the design of better fabrics, I propose to review what has been done to make Orlon a more versatile fiber.

Consider first the versatility derived from high affinity for basic dyes. Basic colors are the brightest colors known, but until recently they have not been used extensively because of their poor light-fastness. Polyacrylonitrile is unique in possessing great resistance to light degradation and in imparting light stability to many dyes incorporated in it. Thus, many basic colors are relatively fast when applied to Orlon, although they fade rapidly when applied to other fibers. While searching for the optimum method to dye Orlon, L. A. Hiller seized upon these facts to point out how much easier it would be to obtain any desired unions or cross-dyeings of Orlon with wool if basic dyes were applied to the Orlon and acid dyes to the wool. Under these conditions the color of either fiber could be varied at will, independently of the dye being applied to the other. Thus began the development of an Orlon with good affinity for basic dyes. The rest of the story is one of fiber modification, dye selection, dye synthesis, and the development of practical commercial methods for the dyeing of all types of fabrics of Orlon.

Fiber modification to impart affinity for dyes can be accomplished only at some cost to other properties; for example, softening point, resilience, or fiber color and color stability. Consequently, a minimum affinity for basic dyes was first imparted to the fiber. Thereafter, affinity was gradually increased as it was found possible to avoid any sacrifice in properties. Figure 1 shows the progress to date toward optimum affinity for basic dyes.

In 1953, light and medium shades were possible on both knit and woven goods at the boil, but dark shades required pressure dyeing. With the enhanced affinity available during 1954, 1955, and 1956, a complete shade range was possible on knit goods at the boil, but only light and medium shades could be obtained on woven goods. Since the more dyeable form became available at the beginning of 1957, an unlimited shade range on all fabrics became possible at the boil. Even rich blacks can now be achieved in 2 hr. of dyeing time at the boil, which gives 6 or 7 hr. for the full cycle.

The progress on shade range is even more dramatic. This is shown in Figure 2, which lists "Sevron" basic dyes for use on Orlon. All of these dyes on Orlon pass an 180° F. wash-fastness test better than vat dyes on cotton. Note the good light-fastness ratings of these dyes; their brightness is of particular interest to us because sweaters are our largest market, and we labor under the phi-

[1] Trademark for Du Pont acrylic fiber.

[2] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

losophy that sweater girls should be *seen*. However, when bright colors are applied to a fiber with poor initial color, one obtains a muddy shade. Consequently, it became imperative to seek means for producing fiber with the best possible initial whiteness.

Pure polyacrylonitrile polymer is snow white. If it were possible to form a fiber from absolutely pure polyacrylonitrile, probably it would be snow white also. Stability to discoloration by heat is also a function of purity; for example, the adsorption of cuprous ions renders the fiber much more subject to discoloration by heat. Polyacrylonitrile is a powerful scavenger for metallic ions.

When polyacrylonitrile is heated at a sufficiently high temperature, it first yellows, then browns, and finally turns coal black. During the initial stages of this sequence the color is nearly pure yellow; consequently, initial whiteness can be evaluated adequately

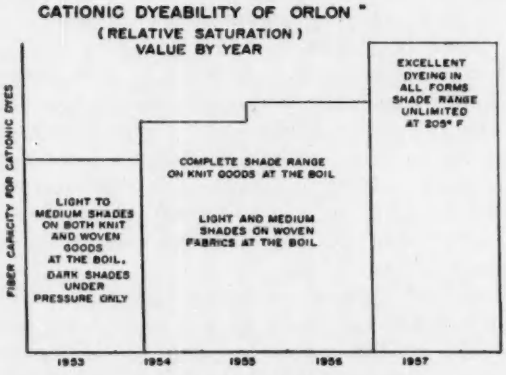
CATIONIC DYEABILITY OF ORLON "
(RELATIVE SATURATION)
VALUE BY YEAR
FIBER CAPACITY FOR CATIONIC DYES
LIGHT TO MEDIUM SHADES ON BOTH KNIT AND WOVEN GOODS AT THE BOIL.
DARK SHADES UNDER PRESSURE ONLY
COMPLETE SHADE RANGE ON KNIT GOODS AT THE BOIL
LIGHT AND MEDIUM SHADES ON WOVEN FABRICS AT THE BOIL
EXCELLENT DYEING IN ALL FORMS SHADE RANGE UNLIMITED AT 205° F
1953
1954
1955
1956
1957

Fig. 1.

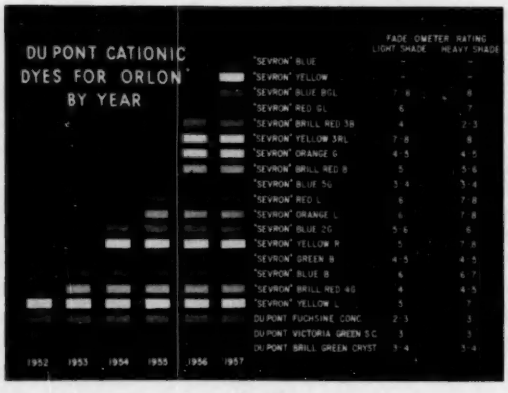

Fig. 2.

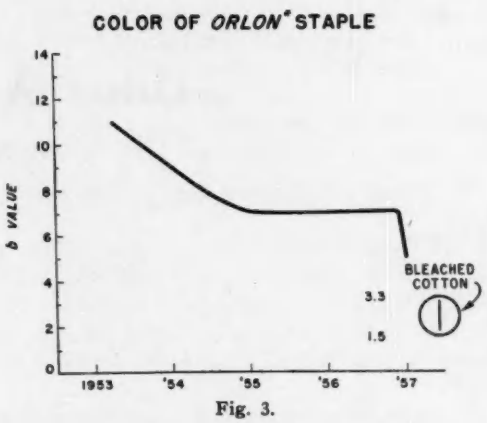

Fig. 3.

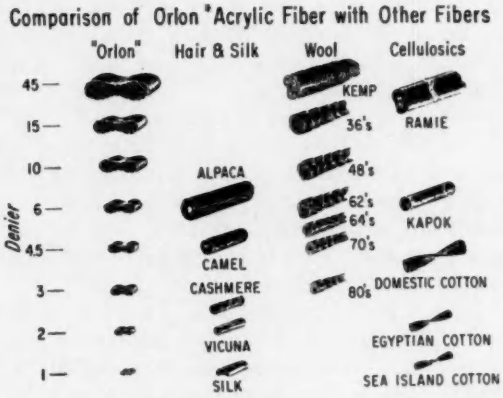

Fig. 4.

by measuring yellowness using a differential colorimeter and expressing the result as a *b* value in Adams coordinates.

In Figure 3 is recorded the progress to date in the drive for better initial whiteness. The Orlon of today is much whiter than unbleached cotton, which often has a *b* value of 9 or 10. Note also that the goal of bleached cotton is only several units away.

The colors recorded were measured on unbleached plant production. Good initial whiteness stemming from fiber purity is a most desirable property because whiteness obtained by bleaching is lost more quickly on exposure to heat. This is illustrated in Table I, which compares the color stability of a recent sample of Type 42 Orlon staple with that of a bleached sample which had poor initial color.

Note that although the early sample could be bleached to *b* value of 3 compared with 5 for the initial color of recent production, after bleaching it

TABLE I. Stability of Color to Heat

	b value	*b* value after heating 20 min. at 165° C.
1957 Type 42	+ 5	+14
Early Type 42	12	28
Early Type 42 bleached	3	21

yellowed much more rapidly when heated at 165° C. than did the unbleached recent production.

Good initial whiteness is vital in fiber which is to be dyed to a pastel shade. For example, Orlon with an initial *b* value of +5 can be dyed to a clear pastel blue, but that with a *b* value of +13 gives only a greenish-blue. If an iron heated to 160° C. is rested continuously on these dyed fabrics for 30 min., the fiber with an initial *b* value of +5 is only slightly discolored, whereas that with an initial *b* value of +13 shows a dark brown imprint of the iron which completely masks any of the blue color.

To make possible still greater versatility in fabric styling, color-sealed black Orlon staple was introduced in 1954. This makes it possible to prepare a wide variety of three–color styles by piece-dyeing the same fabric.

Good initial whiteness and affinity for basic colors make possible good whites and attractive coloration, but this is only the first requirement which a truly versatile fiber must meet. Varieties of the fiber and technology must be available to permit the preparation of widely different types of fabrics with good aesthetic and functional properties.

If every fiber of wool were identical with every other such fiber, wool would not have its reputation for versatility today. Let us compare wools of different fineness with Orlon fibers of approximately equivalent denier.

Three denier Orlon corresponds to the finest lamb's wool, or 80's grade; the 4½ den. staple has a texture like 64's. Similarly, 6- and 10-den. Orlon is comparable with 60's and 48's, respectively. Camel's hair falls in the 3- to 6-den. range, alpaca in the 6- to 10-den. range, and kemp fiber is approximately 45 den. It is interesting to note that no wool fiber equals the fineness of 1-den. Orlon, the latter being comparable to Sea Island or Egyptian cotton or silk. Two denier Orlon is comparable to Vicuna or cashmere. Type 39 staple is a blend of deniers engineered for the woolen system of spinning; its average denier is comparable to 62's–64's wool.

By taking advantage of the wide spectrum of deniers, as shown in the left hand column of Figure 4, it is possible to produce fabrics with textures softer and finer than Vicuna, or with textures equivalent to the coarsest tweeds. Kempy textures and other novelty effects are possible with the heaviest denier. However, new technology was required before full advantage could be taken of these deniers; let us examine the record of advancing technology on the use of Orlon.

The first major breakthrough came with the application of the high-low-shrink principle, which greatly improved the bulk and feel of Orlon sweaters. In fact, it was this principle plus bright dyes which made it the leading sweater fiber. The inclusion of a proportion of high-shrinkage fiber in spun yarns of Orlon was found to cause them to increase markedly in bulk when boiled off.

The same principle can be used to advantage in woven fabrics. In Figure 5 is shown the bulk of a woven fabric after boil-off in water as a function of the percentage of high-shrinkage Orlon used in conjunction with the balance of low-shrinkage Orlon. Note that between 30 and 40% of the high-shrink fiber gives maximum bulk. Control of fabric shrinkage is also easier at this concentration.

As one might expect, the use of a proportion of high-shrinkage fiber causes some shrinkage of the fabric during the boil-off, but in spite of this decrease in area, there occurs the desired increase in specific volume. In Figure 6 is plotted fabric

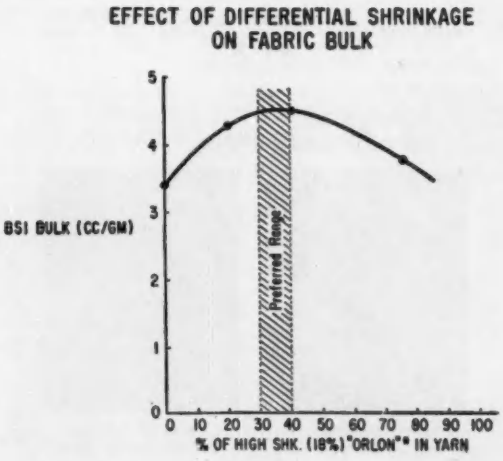

Fig. 5.

shrinkage of 2 × 2 twill, suiting-weight fabrics occurring when the fabrics were given a relaxed boil-off. In this case a concentration of 30% high-shrink fiber caused the fabric to shrink about 15%.

Relaxation of the high-shrink fibers causes them to draw in toward the core of the yarn, whereas those with low shrinkage are thrown toward the surface where they can be seen and felt. This has been demonstrated by spinning yarns from a blend of high-shrinkage fibers dyed red and of low-shrinkage fibers left white. After boil-off the yarns took on a frosted appearance due to the concentration of the white fibers in the surface of the yarn. In this manner it is possible to direct fibers of specific deniers or composition to the surface or to the core of the yarn to give the desired aesthetic and functional properties.

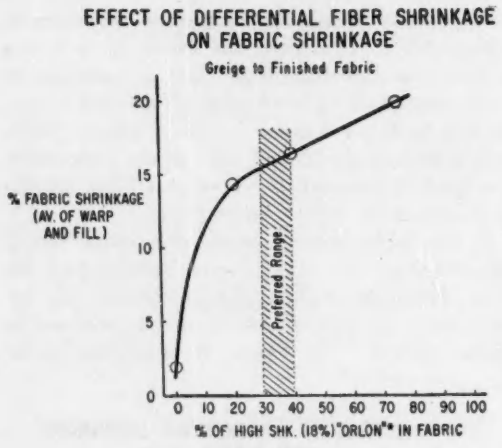
EFFECT OF DIFFERENTIAL FIBER SHRINKAGE ON FABRIC SHRINKAGE
Greige to Finished Fabric
% FABRIC SHRINKAGE (AV. OF WARP AND FILL)
20
15
10
5
0
Preferred Range
0 10 20 30 40 50 60 70 80 90 100
% OF HIGH SHK. (18%) "ORLON"* IN FABRIC

Fig. 6.

IMPROVED COVER THROUGH HIGH-LOW SHRINKAGE PRINCIPLE
55/45 "ORLON"/WOOL NO HIGH-SHRINK "ORLON"*
55/45 "ORLON"/WOOL 30% HIGH SHRINK "ORLON"

Fig. 7.

For example, by using low-shrinkage fibers of low denier, blended with high-shrinkage fibers of high denier, it is possible to spin yarns which after boil-off will have a high concentration of low-denier fibers in the surface and of high-denier fibers in the core. This can be used to produce fabrics having a very soft, luxurious feel due to the low-denier fibers in the surface of the fabric, and improved liveliness due to high-denier fibers present in the core. Conversely, a tweedy feel can be obtained by surfacing low-shrinkage fibers of high denier.

Within certain limits the principle can be extended to blends of other fibers, both natural and synthetic, with Orlon. Aesthetics characteristic of any given fiber can be built into a fabric by causing that fiber to migrate to the surface. A note of caution should be given here, however. If the fiber differs materially in abrasion resistance from Orlon, it is importanct to surface simultaneously a proportion of Orlon of the appropriate denier and to dye the two fibers to similar shades in order to avoid a "frosting" of the fabric which can occur if one of them wears away preferentially.

Use of the high-low-shrink principle has proved a "must" in fabrics where good surface cover is desired. In Figure 7 is shown photomicrographs of two fabrics both made from 55% Orlon and 45% wool; the one with the better cover has been spun from 30% high-shrink and 25% low-shrink Orlon. Note the prominent twill lines and threadiness of the sample containing no high-shrink Orlon.

A further improvement in the surface cover of fabrics from high-low-shrink blends can be achieved by napping the fabrics prior to bulking. This is well illustrated by two woolen experimental flannel-type fabrics which were woven from warp and filling yarns of the composition tabulated in Table II. The average composition of the fabrics was 70% Orlon and 30% wool. The thready fabric at the left in Figure 8, which has poor surface cover, was fin-

TABLE II. 70/30 Orlon/Wool Flannel

Warp yarns:	20% High shrink, 2 den. Orlon 20% High shrink, 3 den. Orlon 60% 80's wool
Filling yarns:	30% High shrink, 2 den. Orlon 30% High shrink, 3 den. Orlon 40% Low shrink, 2 den. Orlon
Finishing:	Napped on a Gessner napper and cropped in the greige. Dolly washed, scoured in a beck at 212° F., sheared, and semi-decated.

ished by a sequence involving fulling, Dolly washing, scouring in a beck at 212° F., shearing, and semi-decating. The second fabric shown at the right, which has good surface cover and a much superior feel, was finished in the same manner except that it was first napped and cropped in the greige. Cover achieved in this manner, which is very stable to washing or normal wear, can be varied over wide limits by using different degrees of napping. Thus we see that the basic technology has been worked out for the production of a wide variety of fabrics with excellent functional and aesthetic properties made from Orlon acrylic and its blends with wool.

There remains the broad field of blends with cotton and rayon. Here the preferred concentration of Orlon in the blend is 80%. One of the bases for choosing this concentration in knitted fabrics of Orlon and cotton is shown in Table III. This records the area shrinkage occasioned by five home machine launderings at 105° F. followed by air drying. Note that jersey fabric knit from singles yarn of 80% Orlon and 20% cotton, not resin-treated, shrank only 2.4% in area, whereas the resin-treated cotton fabric shrank 12% if knit from singles yarns and 9% if knit from two-ply yarns. Note also that a concentration of 80% Orlon is required to accomplish this result, for the 60/40 blend shrank almost 6%.

Orlon in the blend makes possible new and unusual textures. When Orlon staple of the same cross section as that of cotton is used, the blend fabric is characteristically softer than that of all cotton. When using higher deniers of Orlon it is possible to obtain textures permanent to washing, ranging from soft through intermediate to very harsh. Bright Orlon contributes an attractive luster which would otherwise have to be achieved by special processing and chemical treatments; cotton contributes freedom from static.

A blend of 80% Orlon with 20% cotton or rayon is also preferred in woven fabrics to impart the hand, ease of care, and neatness retention characteristic of a quality acrylic fiber. A good measure of ease of care is the amount of work required to iron the fabric. This can be determined if the person stands upon a Lauru platform while doing the ironing. The work performed is recorded automatically. In Table IV is recorded the work expended in ironing a series of comparable lightweight batiste fabrics made from blends of Orlon with cotton and rayon.

If first we examine the data for ironing dry fabric with a steam iron, we note in the rayon series that a concentration of 80% Orlon is required to yield ironing properties closely approximating those of 100% Orlon, which, incidentally, is extremely easy

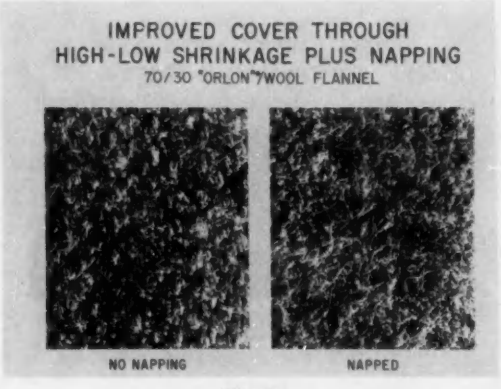

Fig. 8.

TABLE III. Shrinkage of Jersey Fabric on Laundering

	Area shrinkage, %			
	100% Cotton	Orlon/cotton 60/40	Orlon/cotton 80/20	100% Orlon
Singles, untreated	16.9	5.8	2.4	1.7
Singles, tensionless calendered	12.3	5.4	2.7	2.5
Singles, resin treated	12.7	6.0	5.2	1.5
Two-ply, resin treated	9.6			

TABLE IV. Effort Required to Iron a Shirt Back

	Cotton series		Rayon series	
Composition	Effort, kg. × sec.	Appearance* after ironing	Effort, kg. × sec.	Appearance* after ironing
Dry fabric ironed with steam iron				
100% Orlon	35	1.0	35	1.0
80% Orlon	117	2.9	49	1.0
70% Orlon	148	3.5	82	1.5
60% Orlon	200	3.6	94	2.4
50% Orlon	226	4.0	126	3.1
100% Cotton	186	4.5		
Sprinkled fabric ironed with a dry iron				
80% Orlon	76	1.0	47	1.0
100% Cotton	139	1.3		

* Appearance rating scale: 1 = perfect; 3.0 = barely acceptable in appearance; 5 = very wrinkled.

TABLE V. Condition of Batiste Fabrics after Automatic Laundering and Tumble Drying

	Rating*
100% Orlon with anti-stat	1.5
100% Orlon without anti-stat	3.5
80/20 Orlon/cotton	3.5
80/20 Orlon/rayon	2.7

* Subjective rating scale: 1 = perfect condition; 5 = severe wrinkling.

TABLE VI. Condition of Batiste Fabrics after Ironing and Tumbling in Dryer

	Rating*
100% Orlon plus anti-stat	1.1
80/20 Orlon/cotton	3.0
80/20 Orlon/rayon	1.1

* Subjective rating scale: 1 = perfect condition; 5 = severe wrinkles.

to iron. In rayon blends containing less than 80% Orlon the appearance of the ironed fabric was less satisfactory, even though, in the case of the 50/50 blend, nearly four times as much work was expended in ironing the fabric. When the 80/20 Orlon/cotton blend was ironed with a steam iron, its appearance was barely passable (rating of 2.9), whereas the rest of the blends were definitely unsatisfactory. However, all of the cotton-containing blends could be ironed to a good appearance if they were first sprinkled and then ironed with a dry iron.

Although acrylic blends need touching up with an iron after washing and drying, nevertheless their appearance after having been automatically washed and dried is a measure of their ease of care. In Table V are shown subjective ratings of the appearance of three of these lightweight batiste fabrics after having been automatically washed and tumble-dried. Note the relatively good rating of the two blend fabrics and the superior appearance of the rayon blend. Note also that unless an antistat is applied to the fabric of 100% Orlon, it does not come out of the dryer in good condition. This is further evidence of the value of using cotton or rayon in the blend to control static.

In Figure 9 are shown photographs of these same fabrics after automatic washing and tumble drying. Note the presence of characteristic fine-grained mussing in the 80/20 Orlon/cotton fabric.

We believe that the differences in ease of care properties shown between cotton and rayon blends in the above illustrations are real and stem from the fact that cotton actually seems to muss in the tumble dryer. Evidence supporting this explanation is shown in Table VI, which shows subjective ratings of the appearance of these fabrics after they had been ironed to remove all wrinkles and then exposed to tumbling in the dryer without washing. Note the better rating of the Orlon/rayon fabric. Rayon does not muss under these dry, hot tumbling conditions. In Figure 10 are shown photographs of these same fabrics after ironing and tumbling. Again note the presence of fine-grained mussing in the Orlon/cotton sample.

Rayon has other advantages for blending with Orlon. It does not have to be combed prior to blending and requires no bleaching or treatment with strong caustic to remove motes. Since caustic adversely affects the color of acrylics, such a treatment is to be avoided if possible. However, it must not be forgotten that blends of Orlon and cotton yield fabrics differing quite markedly in hand, texture, and

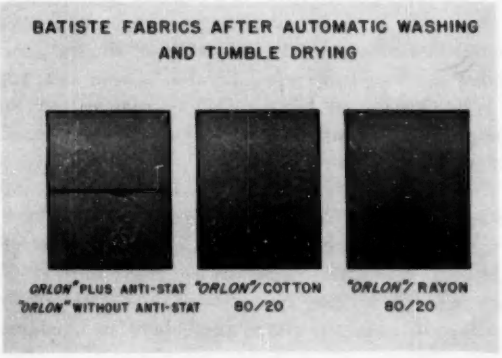

Fig. 9.

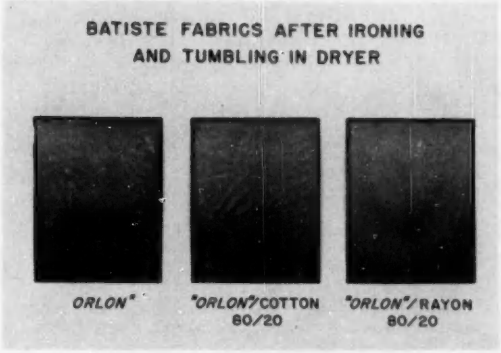

Fig. 10.

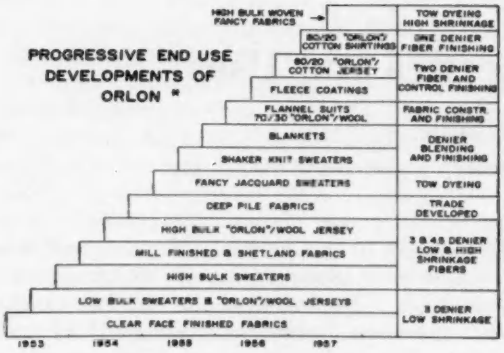

Fig. 11.

luster from those of blends of Orlon with rayon. Consequently one must weigh aesthetic factors as well as functional and economic values in making a choice between these blends.

Methods for continuously dyeing blends of Orlon acrylic fiber with cotton or rayon are under development.[3] Currently we prefer to pad the fabric with selected cationic dyes, using 1 lb./gal. of ethylene carbonate in the pad bath at a pH of 5 with citric acid at 160° F. and then steaming 30–60 sec. at 220° F. Deep shades with excellent light and wash fastness can be so obtained. The cotton or rayon portion can then be dyed with selected vat dyes by known procedures. Procedures are under study which are likely to permit the dyeing of both fibers in one step.

With this last example taken from the burgeoning end-use technology which is adding to the versatility of Orlon acrylic fiber, let us summarize the more important advances; Figure 11 illustrates these. The left-hand column records the year in which the advances were commercialized and the right-hand column indicates how the developments came about. It will be observed that these advances stemmed from properties built into the fiber, notably basic dyeing and good initial whiteness, and from the many different varieties of fiber now available, notably the wide range of deniers, varieties with high and low shrinkage, staple and tow, color-sealed black, etc. They arose from novel yarn and fabric constructions, from the development of optimum dyeing procedures, and the discovery of novel finishing techniques. Progress has been very rapid, because the main consideration which has guided the choice of properties to be built into Orlon and the choice of objectives for end-use research on this fiber has been how best to extend the versatility of Orlon in meeting the needs of the textile industry.

[3] Papers from the Technical Advances Conference, May 1957, p. 53, E. I. du Pont de Nemours & Co., Wilmington 98, Del.

Acrilan[1]—Its Properties and Uses[2]

Walter H. Hindle[3]

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IN ITS suggestion for discussion material, Textile Research Institute raised three basic questions, and in my presentation I hope to answer each query insofar as Acrilan is concerned.

Why So Many Acrylic Fibers?

The first suggested question was "Why is the chemical industry going in so deeply on so many acrylic type fibers?" The significant point in this question is that the question is not "Why is the chemical industry going into acrylic fibers?"; but rather, "Why so many *different* acrylic fibers?".

In answer to "Why so deeply?", it has been well established that acrylic fibers are destined to play an increasingly important part in textile economy. As far back as June 1952, a President's Material Policy Commission forecast that by the year 1975 one billion pounds of acrylic fibers would be manufactured. Market surveys have shown, and are still showing, that in the country's rising economy there is general acceptance of the philosophy of trading upwards; that is, the consumer is not only prepared to pay a little more for better performance but, indeed, demands better performance. The sharply improved living standards of the American and European consumer have brought forth new patterns of living into which acrylic fibers fit admirably.

Shorter working hours with greater emphasis on sports and travel, the emancipation of women from onerous household chores and their greater participation in what has been called a "man's world," coupled with the philosophy of easy living, are all factors which have caused chemical manufacturers to go into the acrylic fibers so heavily.

Another extremely important reason is based on the need of this country to import wool. In 1875 there was one sheep per person; in 1953 there was one-sixth of a sheep per person. It has begun to appear that because of steadily increasing costs of sheep raising in this country, still greater reliance will have to be placed on imports for the nation's wool supply. The events of the past few months have shown how international crises affect the wool market. The obvious long term trend upward in prices of wool, coupled with the fact that historically the man-made fibers have become cheaper as volume acceptance has been reached, is another reason why the chemical industry has gone in deeply for acrylic fibers.

The second part of the question asked by TRI was "Why so many acrylic type fibers?" The answer boils down to competition. It is chemically possible to manufacture a large variety of acrylic fibers because of a fundamental versatility, just as it is physically possible to make a large number of different automobiles. The first entrants into the field developed a patent situation and a consumer acceptance. The companies entering the field later obviously had either to obtain a license and pay royalties to manufacture an existing product or to make a new one. A glance at nylon manufacturing on a world-wide basis reveals the same type of thinking without the flexibility of product possessed by the acrylics.

Properties and Uses of the Acrylics

The second question suggested for discussion by TRI was "What are the properties of acrylic fibers that make them so desirable?" In answering this question, my remarks will be devoted to Acrilan exclusively.

Chemical Resistance

The chemical resistance of Acrilan to acid and solvents is of a very high order. The accompanying chart (Figure 1) shows its relative inertness to a variety of chemical influences. Its resistance to very many other chemicals is equally good, except for caustic alkalies at high temperatures. What does this mean in terms of fiber use? As an example, it

[1] Registered trademark of The Chemstrand Corporation for its acrylic fiber.

[2] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

[3] Associate Director of Research.

has been found that in Monsanto chemical plants, work trousers made of a blend of 50/50 Acrilan/cotton had seven times the wear life of 100% cotton. The initial cost of the Acrilan blend garment was two and one-quarter times that of the 100% cotton. At one time the workers in the chemical industry would not have been inclined to pay the price differential despite the obvious economy, but today the situation is changing, due to the fact that the now well-paid chemical worker is in a position to think more forwardly. Another example of utilization of the resistance of Acrilan to acids and solvents is in automobile fender cloths, where the life of 100% cotton is very short, due to the effects of battery acids and the need for vigorous cleaning.

Bulk in Relation to Weight or Covering Power

One of the valuable characteristics of a material purchased by the pound is a low specific gravity, and this is the case with Acrilan, the specific gravity of which is 1.17. However, more important, in the case of a fiber, is the ability to maintain a bulkiness or high covering power when in yarn form. Acrilan does this because of its tendency to retain its crimp even through textile processing. In addition, the versatility of the fiber makes possible a type called "hi-bulk" which is a stretched fiber and which, when blended with regular Acrilan staple, produces a bulking effect in wet processing. The utilization of this property is widespread; a classic example is provided by jersey knit fabrics made from yarns composed of 65% regular Acrilan and 35% hi-bulk Acrilan. Fabrics of this nature are knit at a weight of 5.9 oz. which, after dyeing and finishing, yield fabrics with a weight of 7.0 oz.

Acrilan does not felt as does wool, but the principle of obtaining greater coverage or a greater density of fabric construction can be very accurately controlled in the case of Acrilan This is an extremely important point to the economy of the jersey knit field. In the case of wool, this is brought about by fulling operations, but with no specific end point as far as shrinking is concerned. Even an extremely prolonged wet treatment of 100% Acrilan jersey goods will not produce bulking to such an extent that the desired ounce weight cannot be finally realized. Wool, on the other hand, still presents a problem in cases where extended wet processing becomes necessary for any reason. The scientific use of the covering power of Acrilan is a very valuable tool in the fabric designer's workshop, and one which is expected to play an increasingly important part.

Warmth in Relation to Weight

Historically, fabric weight has been associated with warmth, but work over the course of the past few years has dissipated this belief. In the case of Acrilan, the blankets, which have received widespread consumer acceptance, prove that body warmth can be maintained without excessive weight. The results of numerous consumer use tests have shown a complete acceptance of the concept that the Acrilan blanket of 3¼ lb. weight provides equal warmth to that of a wool blanket of greater weight. Needless to say, this property is related to bulkiness.

Resilience

There are many definitions of fiber resilience and at least as many tests. However, one practical test in which the resilience of Acrilan, both dry and wet, is compared with that of other fibers is shown in Figure 2. On a practical end use basis, carpets of 100% Acrilan have been field tested in widely dispersed areas over the course of the past four years, and a great deal of information has been developed. A brief summation of these tests shows that Acrilan

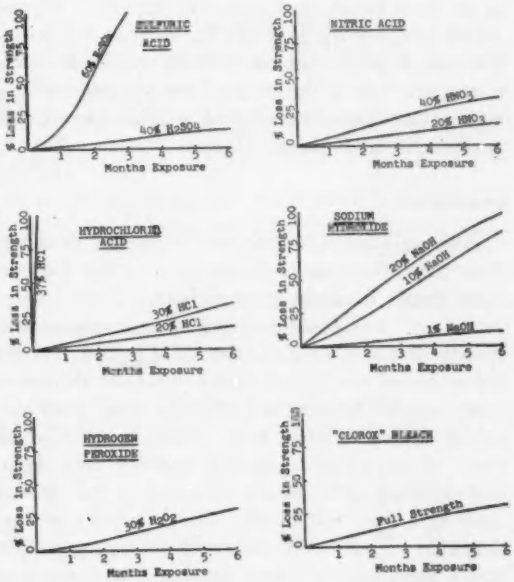

Fig. 1. Effect of chemicals on strength of Acrilan at room temperature.

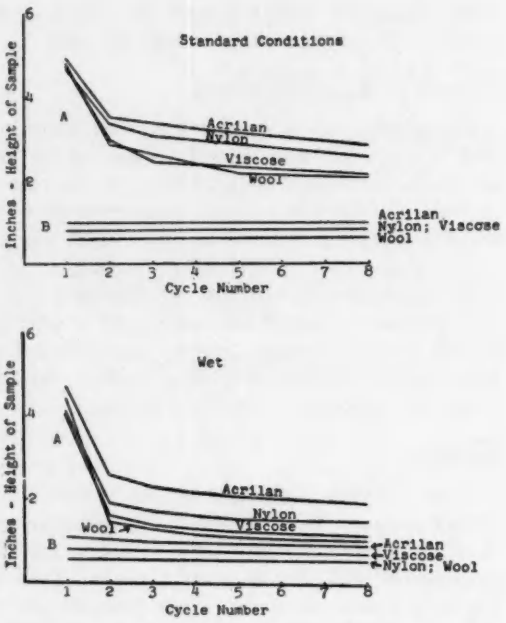

Fig. 2. Compression and decompression of bulk fiber. Diameter of cylinder—2.8 in. Diameter of foot—2.5 in. Wt. of sample—8 g. Sample height at start of cycle—A. Sample thickness under 8-lb. load.

is at least equal, and probably superior, to wool, which has been the previous standard in this respect. Carpets of 100% Acrilan, in both woven and tufted forms, are now in the country's stores, and the property of resilience has played a large part in this major new development.

Aesthetics

Although aesthetic value is a more nebulous term than the definitions of resilience, its chief measurement comes from the cash register. Those "versed in the art" will doubtless agree that cashmere has a luxury of touch which is desirable to many people. By means of the Turbo stapler, a similar delicacy of touch can be obtained from Acrilan tow. Factors of initial denier, stretch ratio, staple lengths, conditions of relaxation, spinning systems, and dyeing and finishing techniques are studied in the development of a very soft hand. The properties of touch desirable in such end use constructions as carpets, men's wear, women's wear, blankets, etc. vary widely, but it is interesting to note that the basic flexibility of the fiber is such as to lend itself to the sciences and arts of the textile trade in widely differing fields of utilization.

Pleat Retention

The ability of fabrics to retain a pleat or crease is extremely important to the basic concept of ease of care, which has come to be extremely important in the textile trade's contribution to philosophies of easy living. Although the majority of the people wish to appear neat, most of us begrudge the trouble it takes to attain this objective. The ease of pleatability and properties of retention, even through washing operations, on the part of Acrilan is a major contribution towards a solution of this dilemma. Acrilan, whether it be in knit or woven form, can be pleated at temperatures lower than those used for many other classifications of fibers. The retention of pleats and creases through washing processes is very important to the busy housewife concerned with the appearance of herself and her family.

Rapid Drying

The rapid drying property of Acrilan is another major factor in ease of living, and the popularity of such a property is well known, especially to housewives and those who travel. Rapid drying is of value in the complete gamut of fabric constructions which require washing.

Sunlight Resistance

Acrilan is extremely resistant to the degrading action of sunlight and weather, and Figure 3 illustrates its superiority over a number of other fibers. This property is of great value in any end use where weather exposure is involved; this has been particularly noticeable on a commercial basis in drapery fabrics of 100% Acrilan.

Dyeability

It has long been recognized that color is the factor that first stimulates interest in the purchasing of textiles, and, although fiber manufacturers have built sterling properties into new products, unless then can be dyed to satisfy the needs of a color-conscious public, many of those properties go unappreciated. The development of the acrylics and their fitting into the pattern of ease of living has imposed a new standard of dyeability requirement. Because of their functionality and longer lasting

properties, it is important that these newer fibers be dyed not only in wide enough ranges of shades to satisfy style and merchandising needs, but also that they be dyed in order to withstand the effects of harder wear which the inherent properties of the fiber give to the consumer.

Only a few years ago no woman would ever think of washing a garment of brilliantly colored wool without a radical loss of color, disregarding the shrinkage problems, but in the case of Acrilan no such problems exist. Consumers may handle the garments in ways best calculated to obtain optimum cleansing without concerning themselves as to loss of color.

The dyeing of any 100% fiber is usually considerably more simple than the dyeing of blended fibers. Within the Chemstrand Corporation, study has been devoted to the dyeing of Acrilan blends by methods similar to those used on the fibers for which the dyes were initially developed. Table I lists types of dyes which can be used on Acrilan, as well as other fibers that may be used with the same dyes. It will be readily understood that by adaptation of techniques, Acrilan blended, for instance, with wool can be dyed in a one-bath method using the same dye for both fibers.

In blends with rayon or cotton, one-bath techniques are available which are similar to standard techniques for the dyeing of such well known fiber blend combinations as acetate and rayon. The dyer may select dyes from a wide range of classifications in relation to end use requirements and cost. The utilization of conventional systems of dyeing has made it unnecessary to resort to special equipment, which is very frequently costly and outside the budget of many smaller concerns, nor has it been found necessary to resort to carrier systems of dyeing with their attendant problems.

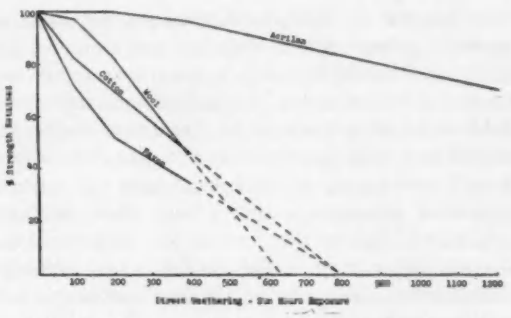

Fig. 3. Direct weathering exposure vs. % strength retained in spun yarn.

The dyeing flexibility of Acrilan has been very carefully planned in the fiber manufacture so as to lend itself to the widest range of end uses, and in this respect can be considered to be unique among acrylic fibers currently in full production. To have a completely dyeable fiber, from a market standpoint, is in itself not sufficient. For styling reasons, there must also be inherent in the fiber the ability to resist dyeing action. Although Acrilan is readily dyeable, it may also be left undyed by the use of blocking agents.

Future of Acrylics

The third and final question suggested by TRI for discussion at this time was "What are the roles anticipated for acrylics in the textile economy of the future?" Again, I will confine my answer to Acrilan.

It has been shown that, in six short years, markets traditionally utilizing wool and other natural fibers

TABLE I. The Dyeing Potential of Acrilan and Other Materials

Dyes	Materials								
	Acrilan	Wool	Rayon	Cotton	Nylon	Acetate	Arnel	Silk	Vicara
Acetate	x				x	x	x		
Basic	x							x	
Neutral premetalized	x	x			x			x	x
Acid premetalized	x	x			x			x	x
Acid	x	x			x			x	x
Chrome	x	x			x			x	x
Soluble sulfur	x		x	x					
Vat	x	x	x	x					
Vat ester	x	x	x	x				x	
Naphthol	x		x	x					

have become available to Acrilan on the basis of aesthetic properties, functionality, and cost. In the early years of development it was necessary to determine to what extent penetration into the textile field could be made without becoming deeply involved in a wide variety of fiber modifications either from a chemical or physical standpoint. It appears logical to believe, now that a basic fiber has been established, that by the devotion of research to the specific rather than to the general, fiber modifications can be made which will fit into specific end use requirements.

Not even the most enthusiastic manufacturers of acrylic fibers believe that their current product has reached the point of finality for all purposes. On the contrary, the Chemstrand Corporation believes that great new and additional markets can be developed for Acrilan because of its flexible base by the engineering of specific fiber properties for specific end uses. All of the nine major properties which have been discussed, and which have been instrumental in developing Acrilan on a widespread basis, are not of equal importance. For example, in an industrial fiber, dyeability might legitimately be sacrificed to yield an increase in tensile strength or abrasion resistance.

New fiber companies, like any other, must make money by gaining acceptance of their product on as wide a scale as possible. One of the methods of expanding the use of acrylics is to devote research time and money to the development of fiber properties that will be more nearly specific to the end use requirements. This having been accomplished, it can be expected that acrylic fibers will have still wider acceptance.

The non-woven field is yet in its infancy, but already the use of fiber is in the scores of millions of pounds; it is not illogical to believe that acrylic fibers will play some important future part.

In conclusion, the accelerated expansion of the use of acrylic fibers, not only in this country but on a world-wide basis, can be expected to justify the predictions made by the Presidential Material Policy Commission in 1952.

Darlan[1]—The First Dinitrile Fiber[2]

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Introduction

The generally accepted classifications of truly synthetic fibers now available are polyamides, polyesters, vinyls, and acrylics. We are now adding dinitrile fibers to this list by discussing some of the interesting characteristics of Darlan, the first dinitrile fiber. We will look at some of the properties of this fiber and attempt to estimate what these properties can mean in terms of fabric performance and market value.

The Monomers and the Polymer

A dinitrile fiber is one prepared from a polymer containing vinylidene dinitrile. This new and interesting monomer was synthesized for the first time in the research laboratories of The B. F. Goodrich Company in 1947 [6]. Its formula is shown in Figure 1.

Notice the difference between the structures of vinylidene dinitrile and acrylonitrile; the alpha hydrogen on the acrylonitrile has been replaced by a nitrile group. The differences in chemical and physical properties between these monomers and between polymers made from them are striking. Vinylidene dinitrile is a clear, colorless liquid which boils at 309° F., freezes at 49° F. [2], and is a powerful lachrymator. The monomer is rather unusual in that it will polymerize by an ionic mechanism at extremely rapid rates [7]. Any source of ions will

[1] Darlan is a registered trademark of the B. F. Goodrich Chemical Company.

[2] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

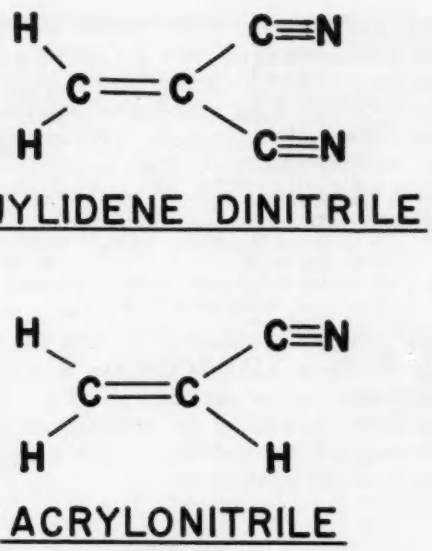

Fig. 1. Comparison of monomer structures.

cause almost instant conversion to low molecular weight polyvinylidene dinitrile. If properly stabilized in the absence of ions, however, the monomer may be stored for long periods of time.

The free radical catalyzed copolymerization of vinylidene dinitrile with other monomers gives striking further demonstration of the uncommon nature of this material [8]. With many comonomers, vinylidene dinitrile shows a strong tendency to form alternating polymers rather than the random polymers often associated with free radical polymerizations. Perhaps Figure 2 will make this clearer.

In this illustration, A and B represent different monomer units in the polymer chain. It is obvious that, although the amounts of each monomer may be the same in each case, the structures of the copolymers are quite different, and polymer properties will probably differ also. The random structure is the one usually associated with addition polymers such as acrylics, and the alternating structure with polymers, such as polyesters, which are formed by a condensation mechanism. Mayo and Lewis in 1944 developed a method for describing numerically the tendency to form alternating polymers in addition polymerizations [12]. The system is based on monomer reactivity ratios. As the product of the reactivity ratios for two monomers in a polymerization approaches zero, the polymer approaches perfect alternation. As this number approaches one, the system becomes completely random.

Random

A–B–B–A–A–A–A–B–B–A–B–A–A–B–B–B

Alternating

A–B–A–B–A–B–A–B–A–B–A–B–A–B–A–B–

Reactivity Ratios

$r_1r_2 \rightarrow 1$ Random
$r_1r_2 \rightarrow 0$ Alternating

Fig. 2. Copolymers of monomer **A** with monomer **B**.

Table I furnishes comparisons of the reactivity ratio products of vinylidene dinitrile and acrylonitrile when copolymerized with other common monomers. Note the strong tendency of the vinylidene dinitrile to form alternating polymers.

TABLE I. Reactivity Ratio Products for Copolymer Systems

Comonomer	r_1r_2 with vinylidene dinitrile	r_1r_2 with acrylonitrile
Styrene	0.0000050	0.0090
2-Chlorobutadiene-1,3	0.000077	0.061
Methyl methacrylate	0.0014	0.24
Vinylidene chloride	0.00059	0.33
Vinyl acetate	0.00059	0.42

The polymer used for the preparation of Darlan staple fiber as now produced is a copolymer of vinylidene dinitrile and vinyl acetate [9]. More complete data for this system is shown in Figure 3. Here again can be seen the strong tendency of the vinylidene dinitrile-vinyl acetate system to form a copolymer with equimolar portions of the two monomers over a very wide range of monomer ratios charged. This is in contrast with the acrylonitrile-vinyl acetate system, where polymer composition is quite dependent upon monomer charging ratios. We therefore believe the structure of Darlan polymer to be as shown in Figure 4—an alternating polymer containing approximately 50 mol per cent vinylidene dinitrile and 50 mol per cent vinyl acetate. We believe this tendency toward alternation is a result of the strong electron attracting force of the two nitrile groups on one carbon atom. This same force should result in high intermolecular attraction between polymer chains by hydrogen bonding. One result of such attraction should be high second order transition points for copolymers of vinylidene dinitrile,

and this is exactly what we find. The second order transition temperature for an equimolar copolymer of vinylidene dinitrile and vinyl acetate is 340° F., about 200° F. higher than for a similar copolymer of acrylonitrile and vinyl acetate. Vinylidene dinitrile, therefore, seems to be a powerful building block with which a whole new family of fiber-forming dinitrile polymers may be produced.

Figure 5 is a schematic flow chart of a method for the preparation of Darlan polymer. The raw materials are natural gas and ammonia. Acetic anhydride and hydrogen cyanide react to form 1-acetoxy-1,1-dicyanoethane, which in turn can be pyrolyzed to vinylidene dinitrile and acetic acid [1]. The acetic acid may be recycled for acetic anhydride manufacture, and the vinylidene dinitrile is then copolymerized with vinyl acetate.

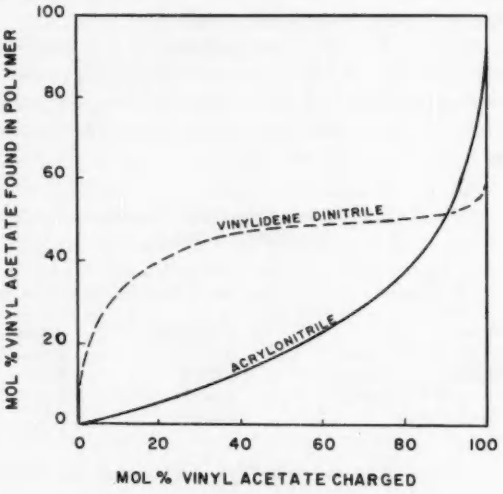

Fig. 3. Copolymer composition curves.

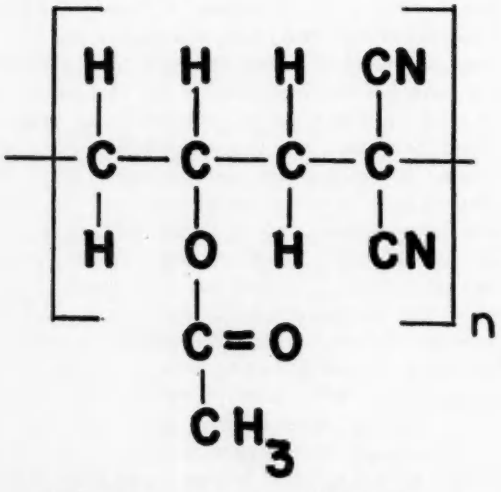

Fig. 4. Suggested structure of Darlan polymer.

TABLE II. Physical Properties of Darlan

Tenacity—dry	1.75 g./d.
—wet	1.5 g./d.
Elongation—dry	25–35%
—wet	25–35%
Modulus	20–25 g./d.
Yield point—stress	0.75 g./d.
—strain	2–3%
Specific gravity	1.18
Moisture regain at 65% R.H., 70° F.	2.2%
Softening temp.	340–350° F.

The Fiber

With a new and interesting polymer which could be dissolved and spun into a fiber, our next problem was the estimation of the fiber's commercial value. Figure 6 shows a typical stress-strain curve for a Darlan single filament. Notice the fiber yields at approximately 0.75 g./den. and 2–3% elongation. Ultimate strength is 1.75 g./den. at 30% elongation.

In Table III will be seen the effect of high temperatures on the tensile strength of Darlan. Note that while the softening temperature of Darlan is not high, the fiber does retain a large part of its original tenacity at temperatures close to its softening point.

Table IV shows the effects of heat and weather exposure on Darlan. All samples were tested at 70° F. and 65% R.H. after exposure; the loss in tenacity both from high temperature and outdoor aging is extremely slight. This was less than the loss of any other fiber tested.

Darlan is probably somewhat less resistant than some acrylic fibers to the action of acids and alkalies. However, the only common textile operation known to degrade the fiber seriously is Kier boiling. It is unaffected by all common dry cleaning solvents, moths, or mildew.

The ease of ignition and rate of flame travel on Darlan fabrics are similar to those of cotton, acetate, and rayon. Life hazards of the volatile combustion and thermal decomposition products are apparently similar to the hazards from silk and wool fabrics.

Darlan fiber as now produced is a difficult fiber to dye. Although the fiber has ample affinity for dyes of some classes, the rates at which many of these dyes diffuse into Darlan are lower than those on many other fibers. To increase the rate of dyeing, dye carriers or high temperatures are used, as will be seen in Table V. The choice of dye class and dyeing method for Darlan depends primarily upon the equipment to be used and the depth of shade desired. In general, as we go toward heavier shades, more severe dyeing conditions become necessary, and piece dyeing requires special precautions to prevent carrier spotting in heavy shades.

TABLE III. Darlan Tensile Strength at Elevated Temperatures

Test temp. °F.	Tenacity (g./d.)	% of 70° F. strength
70	1.75	100
150	1.60	95
200	1.40	80
250	1.20	70
275	1.15	65
300	0.90	50
335	0.70	40
345	0.0	0

TABLE IV. Effect of Heat Aging Upon Darlan

Test temp. °F.	Dry heat aging time (days)	% of orig. strength retained
300	195	100
330	8	100
345	2	95

Effect of Weathering in Direct Sunlight

Exposure	% strength retained
24 mos. in Florida	88
15 mos. in Arizona	100

TABLE V. Recommended Methods for Dyeing Darlan

Shade	Stock or yarn	Piece goods or yarn
Light	Disperse at boil	Disperse at boil
Medium	Disperse at 250° F.; Disperse at boil with MS;* Disperse at boil with *o*-PP; Cationic at 250° F.; Cationic and MS	Disperse at boil with *o*-PP;** Disperse at boil with Latyl Carrier A*** Cationic at boil with Latyl Carrier A
Heavy	Disperse at 250° F.; Disperse at boil with MS; Disperse at boil with *o*-PP; Azoic at 250° F.; Azoic at boil with MS	Disperse at boil with *o*-PP; Azoic at boil with Latyl Carrier A

* Methyl salicylate.
** *o*-Phenyl phenol.
*** DuPont trademark.

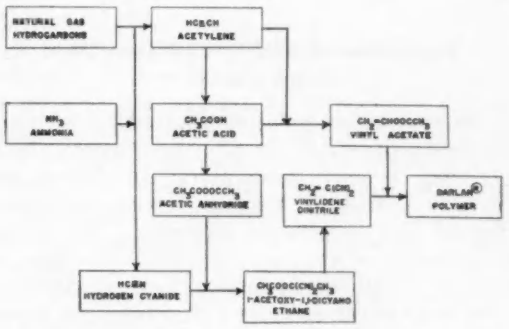

Fig. 5. Darlan polymer via 1-acetoxy-1,1-dicyanoethane.

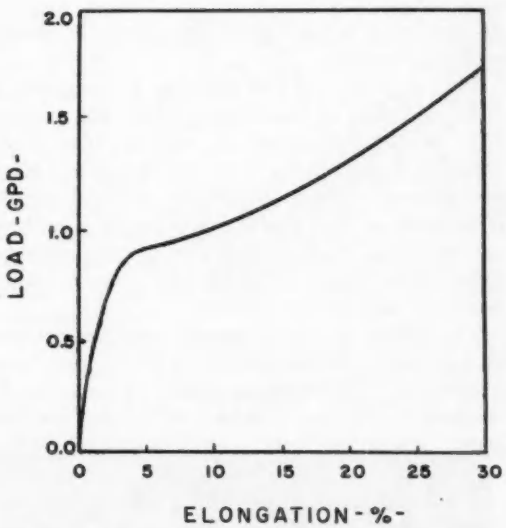

Fig. 6. Stress-strain curve of Darlan staple fiber.

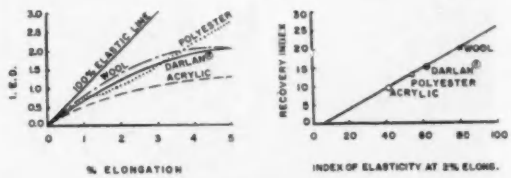

Fig. 7. Elastic behavior of fibers; method of Hamburger, et al.

The fiber presently available from our semi-works plant is slightly off-white. It may be bleached with Textone or hypochlorite; hydrogen peroxide should not be used. Properly bleached Darlan shows no tendency to yellow in the Fadeometer. It may, however, whiten further after 40 Standard Fading Hours.

Predictions of Relations between Fiber and Fabric

Darlan is a fiber with strength, modulus, moisture regain, and other basic physical properties well within the range of useful textile fibers. In addition, the fiber can be dyed, and it appears to have some rather unusual high temperature properties. We wished to determine what these properties could mean in terms of outstanding or unusual fabrics which would be of value to the consumer. Fortunately, in recent years, the textile literature has shown the beginnings of a scientific approach to the difficult problem of fiber-yarn-fabric relationships, with some emphasis on predicting fabric performance from knowledge of fiber properties.

The work of Beste and Hoffman [3], reported in 1950, and that of Hamburger, Platt, and Morgan [11], in 1952, attempted to correlate fiber elastic behavior at low strains with certain types of fabric performance. These publications outlined the determination of fiber elastic properties by two different methods. Both showed strong correlation between fiber elastic behavior and fabric wrinkle resistance and crease recovery.

The method for elastic property measurement used by Hamburger and coworkers is based upon sonic techniques. The immediate elastic deformation determined in this way is shown in Figure 7 for four fibers. All of this work, including that on Darlan,

	TENSILE STRAIN RECOVERY FROM 3% ELONG.	WORK RECOVERY FROM 5% ELONG.
DARLAN®	70	35
ACRYLIC	64	25
POLYESTER	65	30
WOOL	90	54

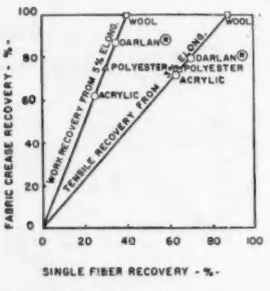

Fig. 8. Elastic behavior of fibers; method of Beste and Hoffman.

TABLE VI. Fiber Properties Affecting Fabric Hand After Beste and Hoffman

	Bending stiffness calc. for 3-d. filaments	Compliance ratio
Wool 70's	180	1.0–1.7*
Polyester staple	180	1.05*
Darlan staple	225	0.89
Acrylic staple	315	0.68*
Silk	657	0.29*

* Literature data.

was carried out by Fabric Research Laboratories, Incorporated. Earlier work in these laboratories had shown a correlation between an index of elasticity, determined from the immediate elastic deformation on fibers, and a composite recovery index, determined from Wrinkleometer data on fabrics. This correlation curve is also shown in Figure 7. The good composite recovery index shown for Darlan is, therefore, a predicted fabric property from fiber data.

Beste and Hoffman measured elastic properties by a cycling technique on the Instron Tensile Tester. Both tensile strain recovery and work recovery from 1, 3, and 5% elongation were measured. They found good correlation between tensile strain recoveries from 3% elongation and work recoveries from 5% elongation with fabric crease recovery. Figure 8 shows our results for four fibers using the Beste and Hoffman technique. The tests were carried out on fibers removed from finished fabrics; our results on the synthetic fibers were generally lower than those reported in the literature. Perhaps this may be due to different sources of fiber (finished fabrics vs. boiled-off staple) and to differences in handling the difficult problem of crimp removal. The single fiber recoveries obtained were plotted on the correlation curves determined by the original workers to give some prediction of expected fabric crease recovery. These data would then predict the possibility of Darlan fabrics with excellent crease recovery.

Beste and Hoffman in the publication mentioned earlier and in another in 1951 [4] discussed the effect of fiber bending stiffness and compliance ratio on fabric hand. They predicted that fibers with low bending stiffness and high compliance ratio would give the fabric hand associated with worsted type materials from wool. The bending stiffness values shown in Table VI are calculated by multiplying the

initial tensile modulus by the square of the denier. This is an approximation assuming round cross-sections. Since neither the Darlan nor the acrylic fiber had cross-sections even approaching a circle, fabrics from both would probably be softer than indicated here. As a matter of fact, extreme softness has been one of the most noticeable properties of all the Darlan fabrics we have prepared. The compliance ratio as used here is the average rate of change in compliance between 5 and 10% elongation. These data indicated to us that Darlan fiber should be considered for wool-like rather than silk-like fabrics.

One of the most difficult fiber-to-fabric relationships to estimate is durability. Hamburger [10] in 1945 published work on a correlation between fiber tensile properties and fabric durability as measured by the Taber Abraser. An energy coefficient was determined by cycling techniques on an Instron Tensile Tester for several fibers. These coefficients correlated with Taber abrasion data on fabrics. The energy coefficient determined for Darlan indicates that Darlan fabrics could show durability considerably above that of rayon, far below that of nylon, and slightly below that of the acrylic fiber tested. In Figure 9 are plotted data for rayon and nylon as shown in Figure 6 of Hamburger [10], with added data for acrylic and Darlan fibers furnished by Fabric Research Laboratories.

Two of the properties of Darlan we have studied are primarily concerned with fiber or polymer fine structure. These are crystallinity and second order transition point. The X-ray diffraction pattern in Figure 10 shows complete absence of crystallinity and perhaps very light evidence of orientation. The second order transition, according to Alexander Brown's publication [15] in 1955, is the melting point of the amorphous phase of a fiber. Table VII compares the second order transition temperature of Darlan with those of three other fibers. It should be recognized that this is not a sharp melting point, and the temperatures given are approximate. Taken together, the results on crystallinity and second order transition can provide us with more indications of fabric properties.

TABLE VII. Second Order Transition Temperatures

Darlan	340° F
Acrylic	175° F.*
Polyester	175° F.*
Wool	>265° F.* (dec.)

* Literature values.

Since Darlan shows no crystallinity, it has no first order melting point, so its maximum useful temperature is in the range of its second order transition. This means a maximum safe ironing temperature of 325–350° F.; this could be a borderline property for apparel uses. This rather low ironing temperature alerted us to watch for difficulty in this area during further fabric development work. On the other hand, the high second order transition temperature of Darlan (both wet and dry) should mean fabrics with excellent dimensional stability and resilience at elevated temperatures such as are encountered in dyeing, washing, drying, and certain industrial uses.

We may summarize our laboratory work by saying that Darlan appears to be a soft, resilient, stable, moderately strong, and durable fiber which gives

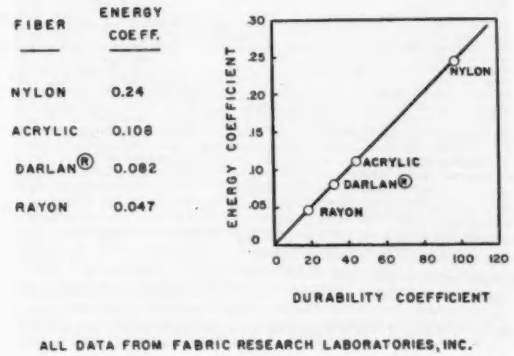

Fig. 9. Estimation of fabric durability from fiber tensile properties.

Fig. 10. X-ray diffraction pattern of Darlan.

TABLE VIII. Elastic Properties of Yarns

	Tensile g./d.	Elong., %	Elastic rec. from 3% elong., % 0.1 sec.	60 sec.
100% Pima cotton (50/1)				
As spun	2.3	4.6	66	77
Steamed	2.1	4.4	61	68
65/35 Darlan/pima (46/1)				
As spun	1.3	4.7	78	94
Steamed	1.1	3.8	88	92
80/20 Acrylic/pima (50/1)				
As spun	1.6	8.1	75	89
Steamed	1.4	7.8	73	85
65/35 Polyester/pima (44/1)				
As spun	1.4	12.0	65	85
Steamed	1.3	11.5	75	95

promise of producing yarns and fabrics of real commercial value.

Fiber-to-Yarn Properties Translation

In our early work on yarns, it soon became clear that resilient fibers do not necessarily make resilient yarns. Our problem became one of the translation of fiber properties to yarn properties. High bulk yarns may be considered as an example of this problem. Our first attempts to prepare high bulk yarns were by the differential shrinkage method used on some other fibers. But Darlan was so stable in boiling water that we just could not get enough shrinkage. In the search for another method for achieving bulk, we hit upon the idea of crimp setting the Darlan fiber. If the fiber is crimp set under steam pressure, the crimp becomes more permanent. Fibers in yarns spun from crimp-set Darlan appear to have lost their crimp. However, a few minutes in boiling water or even a few weeks at room temperature will cause the fibers to revert to their crimped state, giving a lofty high bulk yarn. An example of such a yarn before and after boiling in water is shown in Figure 11.

In Table VIII we show elastic properties of some yarns spun on the cotton system from blends of various fibers with cotton. These data are from a very limited study and probably do not represent the best results that could possibly be obtained with any of these fibers. The test was run by extending 10-in. lengths of single end yarns at a rate of 1 in./min. to 3% extension and immediately releasing the load. Yarn lengths were measured both immediately and after one minute recovery.

Fabric Development

The first commercial success using Darlan was a knitted back deep pile fabric. The pile of this fabric was 100% Darlan. Since the Darlan was all on the face of the fabric, there were no yarn construction problems, and the fabric demonstrated the properties anticipated from fiber data. The pile was soft and yet resilient, with little tendency to mat. The fabric was less susceptible to damage in dry cleaning than fabrics of this type made previously. This is as predicted by the high second order transition temperature and excellent elastic properties of Darlan fibers.

Work with Darlan in cotton blend fabrics is currently under active development; the results to date indicate we can expect soft, wrinkle-resistant fabrics. Laboratory and wear tests on the Darlan-cotton blend fabrics show that in certain constructions, pilling and roughening are a less severe problem than with similar fabrics previously available. Although the fabrics feel soft and delicate, durability is proving completely satisfactory in wear and commercial launderings.

The development work on Darlan in the worsted and woolen goods fields has so far been rather limited. From the fabrics that have been made, we can conclude that Darlan–wool worsted suiting materials have good wrinkle resistance, excellent crease

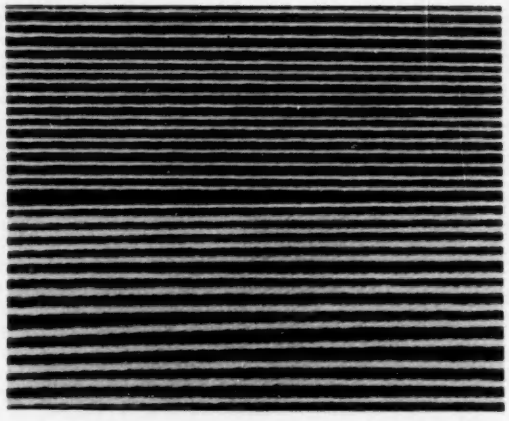

Fig. 11. Yarns spun from crimp-set Darlan. Top: before treatment in boiling water. Bottom: after treatment.

retention, good durability, and virtually complete lack of pilling. Much development remains to be done to translate the Darlan aesthetic values into these worsted fabrics. The difficulties in union dyeing Darlan–wool blends has slowed this work considerably. The woolen type fabrics do show the Darlan soft, luxurious hand. 100% Darlan fabrics are being evaluated in women's coats and suitings.

In the knit goods field, the 100% Darlan crimp-set high bulk yarns are being evaluated primarily in women's sweaters. These sweaters have a remarkably soft yet wool-like hand. Their waistband elasticity is much like that of 100% wool sweaters. Their ability to survive repeated machine washings and dryings is excellent.

We have recently had some success in converting Darlan tow into bulky yarns by way of the Turbo-Perlok system. The differential shrinkage attained is considerably less than that of the acrylics, but it is apparently enough to give a somewhat lofty yarn. Sweaters knitted from these yarns have a silky, soft hand, and they stay soft even after repeated washings.

Merchandising Program

B. F. Goodrich Chemical Company has announced no decision as yet concerning their plans to enlarge the present semi-works production to a full scale commercial plant. The present selling prices for Darlan staple fiber start at $1.85/lb. The present merchandising program is concentrated on developing those specific applications where the Darlan fiber properties can be translated into unique fabrics.

In summary, in Darlan staple fiber we have a new composition of matter, the first of a new class of textile raw materials—the dinitrile fibers. We have reported on some of our determinations of the properties of this fiber. We have further attempted to predict possible fabric properties from these laboratory data, as well as reporting the results of our first fabric development work to substantiate the laboratory predictions. Based on this work, we believe that Darlan dinitrile fiber will fill a definite need for a special purpose prestige synthetic fiber.

Acknowledgment

It is impossible to give individual credit for work done in a project of such broad scope. The work behind the results reported here was carried out by many people at the Research Center of The B. F. Goodrich Company in Brecksville, Ohio and at the Development Center of B. F. Goodrich Chemical Company, Avon Lake, Ohio. Credit for this work belongs to all of them.

Literature Cited

1. Ardis, A. E., U.S. Patent 2,476,270 (1949).
2. Ardis, A. E., et al., *J. Amer. Chem. Soc.* **72**, 1305 (1950).
3. Beste, L. F., and Hoffman, R. M., TEXTILE RESEARCH JOURNAL **20**, 441 (1950).
4. Beste, L. F. and Hoffman, R. M., TEXTILE RESEARCH JOURNAL **21**, 66 (1951).
5. Brown, A., TEXTILE RESEARCH JOURNAL **25**, 891 (1955).
6. Gilbert, H., U.S. Patent 2,514,387 (1950).
7. Gilbert, H., et al., *J. Amer. Chem. Soc.* **76**, 1074 (1954).
8. Gilbert, H., et al., *J. Amer. Chem. Soc.* **78**, 1669 (1956).
9. Gilbert, H., and Miller, F. F., U.S. Patent 2,615,866 (1952).
10. Hamburger, W. J., TEXTILE RESEARCH JOURNAL **15**, 169 (1945).
11. Hamburger, W. J., Platt, M. M., and Morgan, H. M., TEXTILE RESEARCH JOURNAL **22**, 695 (1952).
12. Mayo, F. R., and Lewis, F. M., *J. Amer. Chem. Soc.* **66**, 1954 (1944).

Inertness, Covering Power, and Thermal Pliability Characterize Dynel[1]

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DYNEL, Union Carbide's unique acrylic fiber, first introduced to the trade in 1950, is made in 60–40 ratio from vinyl chloride and acrylonitrile. The polymer is solution spun, using acetone as the solvent. Produced by a comparatively simple process from such low cost raw materials, Dynel holds an enviable potential economic advantage.

Dynel is currently manufactured in fiber sizes of 2, 3, 6, 12 and 24 den. and in staple lengths of 1¼ to 5 in. It is also available as 180,000 total den. tow. The natural fiber is a light cream color and has a specific gravity of 1.30. In many of its physical characteristics and in range of dyeability, it is similar to the other acrylic, vinyl, and dinitrile fibers. Like them, too, it has excellent resistance to mildew and fungus, and to attack by insects such as moth larvae and carpet beetles.

Physical Characteristics

In the 3-den. fiber, Dynel's tensile strength is 3 g./den. with about 35% ultimate elongation. In Figure 1 a typical stress–strain curve is shown in comparison with some well–known non-acrylics. It will be noted that the curve indicates an average stiffness of 8.3 g./den., elastic stiffness or stiffness modulus of 30 g./den., and toughness index of 0.53 g./den. In Figure 2 the relationship between filament denier and tenacity is illustrated. Note the lower unit strengths in the higher denier fibers; less efficient molecular orientation in these bigger filaments is a factor here.

Dynel staple fiber is not a definitely crystalline material, and does not have a sharp melting point. In Figure 3 the stiffness is plotted against temperature in comparison with the same non-acrylics shown in Figure 1. The curve for Dynel shows a second order transition temperature in the range of 80–100° C. As the amorphous phase softens, two things happen: resistance to distortion by external stress or internal strain is markedly reduced, and the fiber becomes susceptible to penetration and swelling by water, a phenomenon of importance in its dyeing and finishing. As temperature goes up, unrestrained Dynel will begin to shrink, relieving residual internal strains; the amount of shrinkage depends on the maximum temperature reached and the time of exposure. Dynel which has been so treated is thereafter dimensionally stable to similar conditions of exposure. Residual strains from inadvertent stretching during yarn and fabric manufacture are relieved in this manner, unless tight fabric construction prevents shrinkage from taking place. Dyeing at the boil, boiling off, or exposing to 250° F. dry heat under conditions of minimum tension are the procedures most generally used.

Figure 4 illustrates the dimensional changes exhibited by Dynel yarns and fabrics heated in air under several different load conditions. The 1 g./1000 den. load curve shows that under minimum tension Dynel is stable up to 250° F.; above 265° shrinkage becomes quite pronounced. Under medium

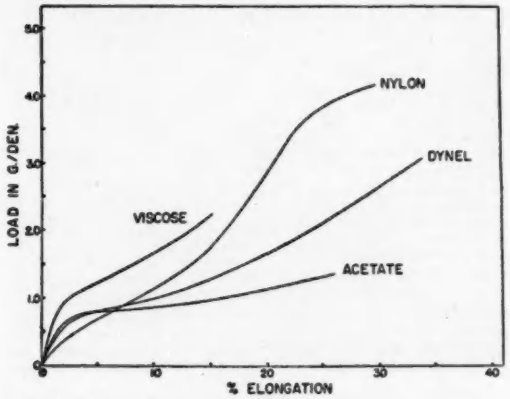

Fig. 1. Typical load-elongation diagram of fibers.

[1] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

load, such as in a tenter frame, Dynel's performance is illustrated by the 10 g./1000 den. curve. Here the tension starts to stretch the Dynel as 200° F. is approached; stretching continues until a peak value is reached at about 250°, at which point shrinkage forces start reversing the action. Near 300°, the initial stretch is entirely overcome. With heavy loads such as might be encountered in a loop drier Dynel will continue to stretch with increased temperatures, as the 20 g./1000 den. curve shows; here the released internal strains are insufficient to overcome the external stresses.

Figure 5 illustrates Dynel's irregular, ribbon-shaped cross section, which can also be detected in the longitudinal fiber views (Figure 6). This shape is responsible for its great covering power and extreme softness, and to a certain extent its rapid wicking and wetting characteristics.

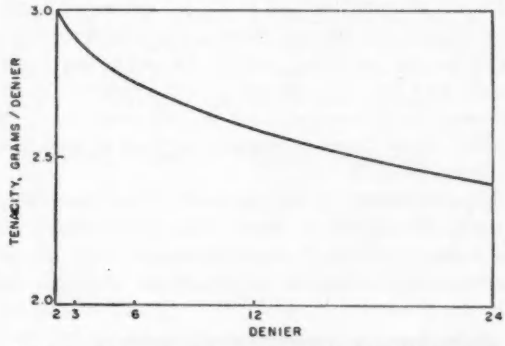

Fig. 2. Fiber strength as a function of fiber denier.

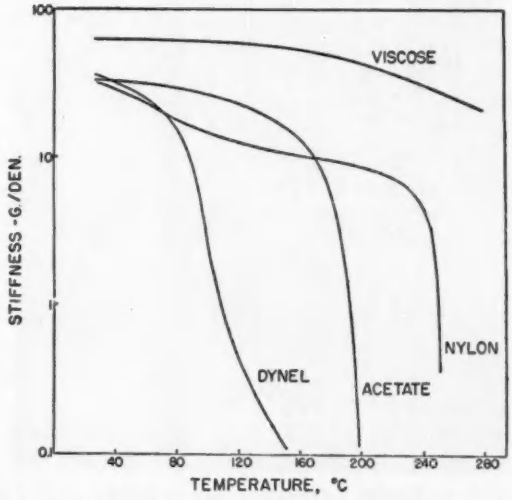

Fig. 3. Ratio of stiffness to temperature for several fibers.

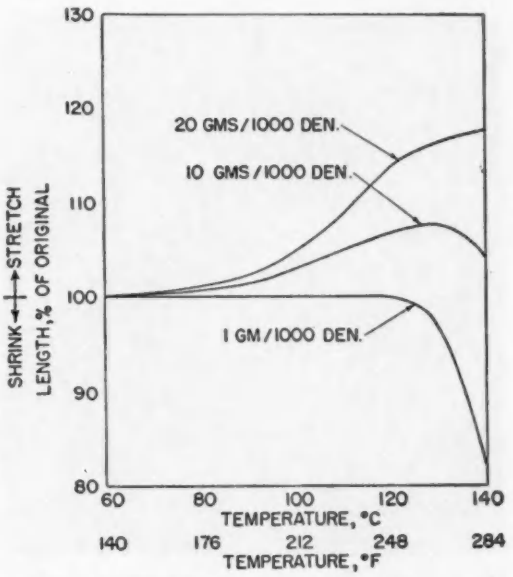

Fig. 4. Dimensional change of typical Dynel yarn vs. temperature under fixed loads (yarn temperature elevated 3° C./min.).

Chemical Inertness

Dynel excels particularly in its general inertness. It is unaffected by strong detergents and soaps and a wide variety of inorganic acids, bases, and salts. It is unaffected by hydrocarbons, dry cleaning solvents, and most other organic chemicals. Acetone, certain cyclic ketones, and some amides and amines exert solvent or swelling action, particularly at higher temperatures. Some aromatic compounds and hot oxidizing reagents also affect the fiber, but on the whole Dynel may be considered to be among the most chemically inert of all the synthetic fibers. Paradoxically, despite such high chemical resistance, dyeing of the fiber is readily accomplished by simple straightforward methods to give a full range of shades.

At ambient temperatures the fiber is impervious to water; the extremely low moisture regain is only 0.4% under standard test conditions. As a result, its wet and dry properties are substantially identical, although in fabric form changes in inter-fiber and inter-strand frictional characteristics due to wetting may result in altered response to mechanical action.

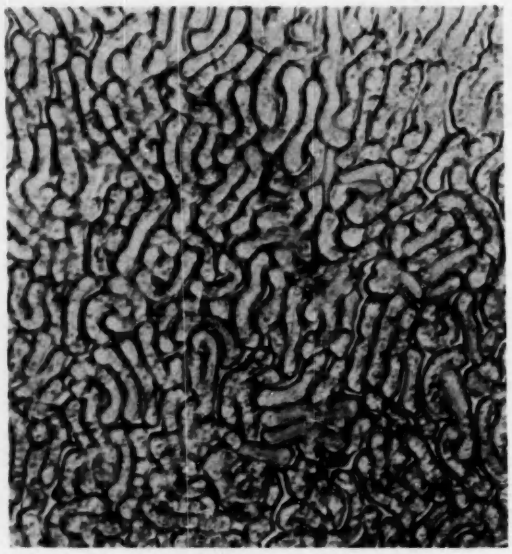

Fig. 5. Dynel cross section; magnification 500 diameters.

Characteristics axiomatically associated with low moisture content in a nonconductor of electricity are a tendency to develop static charges and an inability to dissipate them innocuously; with Dynel this problem can be adequately controlled in various ways, including the use of proper temporary and/or durable antistatic finishing agents. An equally axiomatic attribute, of course, is very rapid drying of extracted Dynel fabrics.

Dynel's inertness gives it another important advantage over other related fibers in its very marked resistance to ignition by such common relatively low-temperature ignition sources as burning matches and cigarettes.

The fiber's composition not only gives it a higher ignition temperature, but also tends toward self-extinguishment upon removal of high temperature heat sources. When forced to burn, Dynel does not melt or drip, but chars; a blanket of this fiber in a Charleston home charred but did not ignite when a child accidentally draped one end against a radiant gas heater while building a play tent in the living room.

This brief resume of Dynel properties would not be complete without mentioning fiber crimp and finish, those attributes imparted during manufacture which are all-important to good fiber processing in any textile application. Carbide applies crimp and finish to Dynel as believed best for optimum processability on the yarn system for which the fiber is intended. Dr. Brown's work [1] has been of value to us in this respect.

Fields of Dynel Usage

One major field of Dynel usage is based primarily on chemical inertness. Filter fabrics have been a solid and unspectacular market, steadily growing; related uses include dust fume bags, air filters, anode bags, and battery separators. In work clothing all-around resistance to attack is provided both in 100% Dynel and in cotton blend fabrics. In paint rollers the fiber's wet resilience and easy clean-up are as important as solvent resistance; full exploitation of the water-base and latex-type paints has only been attained through the use of Dynel rollers. Despite the inroads of cheaper blends, the all-Dynel item remains the premium roller. In wash mitts and applicators for wax emulsions, wet strength and resilience in alkaline solution are all-important. In fender cloths Dynel is resistant to battery acid and to grease and oil.

One new and interesting use is that of a veiling or overlay material in the manufacture of low pressure glass reinforced resin laminates. The Dynel overlay may be applied to either one or both sides

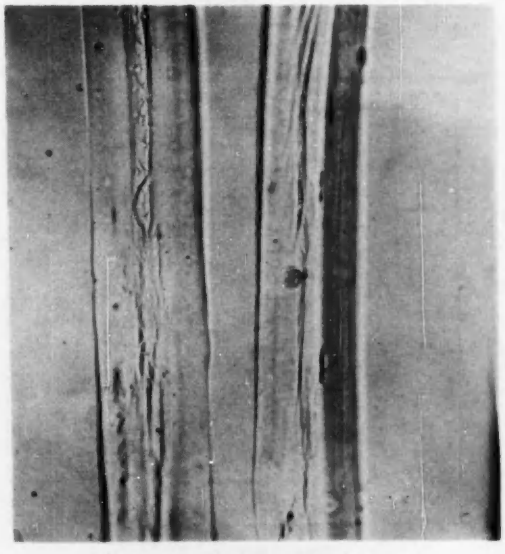

Fig. 6. Longitudinal view of Dynel; magnification 500 diameters.

of the laminate, in the form of a woven, knitted, or non-woven fabric. The Dynel structure wets exceptionally well with resin, resulting in a resin-rich surface. This improves the chemical and abrasion resistance, and imparts a smoother surface. End products being manufactured from such laminates include chemical tanks and pipes, fume ducts, boats, etc.

A second field of Dynel usage is based primarily on its flame resistance. Although this is a complicated field in which fabric construction, dyes and finishes play an interrelated part, numerous fabrics of Dynel alone have passed the rigid requirements of the New York Board of Standards and Appeals and similar agencies. The draperies and bedspreads on the liners S.S. United States and S.S. America are 100% Dynel, selected for safety reasons. Dynel is also used in trim fabrics for interiors of airplanes, in blends with Saran and Fortisan in such items as casement cloths, in military applications, and other uses where resistance to ignition is important.

Uses Based on Thermal Pliability

The biggest markets for Dynel during the past few seasons have been those in which its unique thermal characteristics are of primary importance. By precise control of tension or pressure during the application of wet or dry heat to various types of fabric for proper periods of exposure, a wide range of effects is obtained. In pile fabrics for apparel uses Dynel is an ideal face fiber, and is widely used both 100% and in blends (largely with Orlon). Although luster, coverage, and resilient softness are important plus factors for Dynel in this fabric, the principal advantage is the ease with which a beautiful appearance can be obtained in the final hot finishing operations. An important related use is in the backing yarn of knit pile fabrics. Here, the Dynel backing is so treated that the plain knit construction becomes stabilized dimensionally; thereafter it can be cut without ravelling and handled without distortion.

Another way in which the thermal pliability of Dynel is utilized is in the making of shaped articles, such as summer hats. These hats are made of flat woven fabrics that are drawn and set in heated metal molds. Dynel fabrics which have been stiffened thermally in this manner are utilized in various special applications, such as cap visors, especially for military uses.

Uses Based on Other Properties

Another field of application is based on its excellent wet stability, warmth, and attractive hand. Dynel by itself combines many of the characteristics of the more precious natural fibers with a number of man-made features not available in any natural fiber. By blending with the older fibers, many fabrics of exceptional interest have been obtained.

The amount of Dynel in apparel blends ranges between 25% as a minimum to realize appreciable benefit and 50% as a maximum in order to avoid possible damage by improper ironing and pressing. In blends with cotton for underwear and children's sleepers, 25% Dynel provides light-weight warmth, keeps the cotton soft on repeated laundering, and cuts shrinkage in half. In blends with wool, particularly in soft fabrics with 70% wool, Dynel's crease retention, good coverage, vicuna-like hand, and resistance to pilling have resulted in truly excellent men's wear fabrics of increased durability and wear life. In rayon and rayon–acetate blends, 25–35% Dynel imparts crease retention, abrasion resistance, washability, and various novelty and hand effects; these blends are used in women's suiting fabrics and in boys' slacks for increased durability.

Dynel Spun with Color

Regular Dynel in its natural color has been supplemented by the development of several modified forms. The first of these was dope-pigmented Dynel, or Dynel spun with color. There are 11 different hair-like or fur-like colors now available. Not only does Dynel make an excellent pile face fiber for the synthetic fur trade, but in 24 den. it provides very life-like hair for chignons, wigs, doll hair, etc. For the latter application Dynel is furnished as uncrimped tow on spools.

High Bulking Dynel

Recently we introduced a high bulking fiber, Type 63. This fiber exhibits 25% shrinkage in boiling water compared to 2.5% shrinkage for regular Dynel. It can be used to bulk any non-shrinking fiber by either wet or dry techniques. Principal field of application has been in cotton blends where 25% does an even better job than regular Dynel in upgrading the quality of winter underwear and children's sleepers. In laboratory tests the unit

weights of all cotton, cotton–regular Dynel, and cotton–Type 63 Dynel fabrics required to give an equal warmth were found to be 4, 3, and 2, respectively, showing how accurately the term "warmth without weight" describes this blend. Type 63 is also used in pile fibers and in backing yarn for certain novelty effects.

Carpet Dynel

An even more significant development is the introduction this year of Type 77 Dynel for carpeting. This fiber, while of the same chemical composition as regular Dynel, has distinct physical properties of its own that result from a round cross section and a different method of fiber production. This fiber is being made in 18 den. and can be spun on both of the conventional carpet yarn spinning systems. Its properties have been designed to make a soil resistant, durable, and resilient fiber that closely resembles carpet wool in hand and appearance. Details of the properties required have been developed with the cooperation of carpet mills dating back to a joint research agreement made with one mill in September, 1952.

Carpet Dynel retains the inherent characteristics of resistance to burning, molds, beetles, and chemicals; dyeability to fast colors; and thermoplasticity for twist setting and retention. Because of its physical nature, however, the fiber produces a lofty yarn with a pleasing, non-slippery, wool-like hand and high covering power. It has been made in a wide variety of carpet constructions. Many of these have been wear tested, with other materials for comparison, for long periods of time under a wide variety of traffic conditions—in homes, offices, and industrial plants, in cafeterias, and on stairways. The wearing qualities, soil resistance, ease of cleaning, and retention of texture proved so favorable that Dynel carpeting was introduced by three mills at the Chicago Home Furnishings Show in January.

Dyeing Dynel

Methods used for coloring Dynel are simple and straightforward. The disperse, neutral dyeing premetalized, and cationic dyes constitute the major groups of colors applied to Dynel. They are applied at temperatures near the boil to give a full range of colors of acceptable fastness properties. In addition, the acid dyes are readily applied in a wide range of colors by the cuprous ion method. This fundamental dyeing process, originally announced at the 1950 Annual Meeting of the Textile Research Institute [2], is now covered by U. S. Patents [6] owned by Union Carbide Corporation.

There are three basic requirements for the successful dyeing and finishing of Dynel: first, the use of dyeing temperatures above 205° F., second, restoration of the luster of the dyed fiber, and third, consideration of the fact that the fiber is thermoplastic. Temperatures above 205° F. are essential because it is only within that range that the fiber's moisture absorption becomes high enough to be of practical

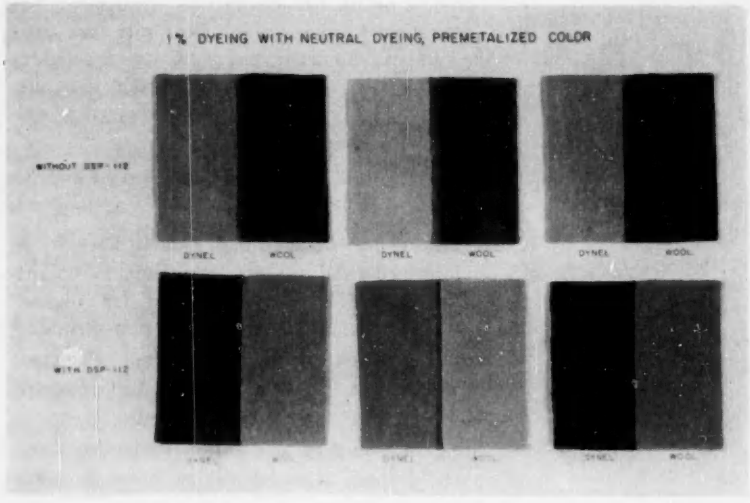

Fig. 7. Effect of DSP-112 in the dye bath. Top, without; bottom, with.

value in dyeing. When the dyed fabric is cooled again to ordinary temperatures, the color is fixed in the fiber and rendered unavailable to the action of water and the various chemical agents that might be expected to discharge or alter the color.

When Dynel absorbs water at 205° F. or above, it tends to deluster. This has a profound effect on the shade attained. Since light fastness is markedly better on lustrous fiber than on dull fiber, it is best to bring the dyed material up to its full color value by restoring the luster. This can be accomplished with dry heat at 240° F., by semi-decating with steam for short cycles, or by using a boiling sodium sulfate solution.

New advances are constantly being made in improving and simplifying the methods used for dyeing Dynel. Particularly noteworthy has been the recently announced commercial method for dyeing under superatmospheric pressure, whereby improved color yields and shorter dyeing cycles are realized [4, 5].

New Dye Carrier

Another advance of equal or even greater significance has been the development of a new dye carrier for Dynel known as DSP-112. This new product, now commercially available and used to the extent of 2–3% of the weight of fiber being dyed, is easily emulsified and is particularly effective. Its great value lies in the fact that small amounts increase the affinity of Dynel for dyes to such an extent that in blends with wool, for example, certain colors normally having higher affinity for wool can be made to have a higher affinity for Dynel instead. This makes possible excellent union shades for Dynel and wool in a single bath one-stage dyeing. For example, by employing neutral dyeing premetalized dyes for the Dynel and acid dyes for the wool, union shades with excellent fastness are realized. Union shades in blends of Orlon [2] and Dynel are likewise possible with the cationic dyes by employing DSP-112 to increase the affinity of Dynel for this class of dyes to the level of that of Orlon.

Several pieces of wool and Dynel in pairs are shown in Figure 7. Each pair was dyed in one bath. At the top are shown the relative affinities of these two fibers for three neutral dyeing premetalized dyes. In each case note the relatively high affinity of the wool for the dyes. At the bottom similar dyeings of wool and Dynel were made with 3% DSP-112 in the bath. Note this time that the Dynel is much heavier than the wool. Acid dyes may be used to adjust the shade of the wool with little or no staining of the Dynel.

Durable Auxiliary Finishes

In the field of synthetics, the essentially hydrophobic fibers are susceptible to the development of objectionable charges of static electricity. Many materials are successful in preventing the develop-

[2] Du Pont's trademark for its acrylic fiber.

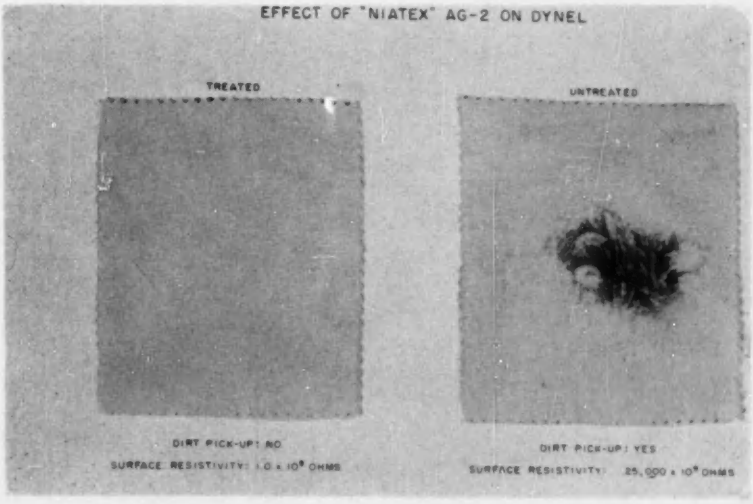

Fig. 8. Treated and untreated Dynel fabrics after 20 machine washes.

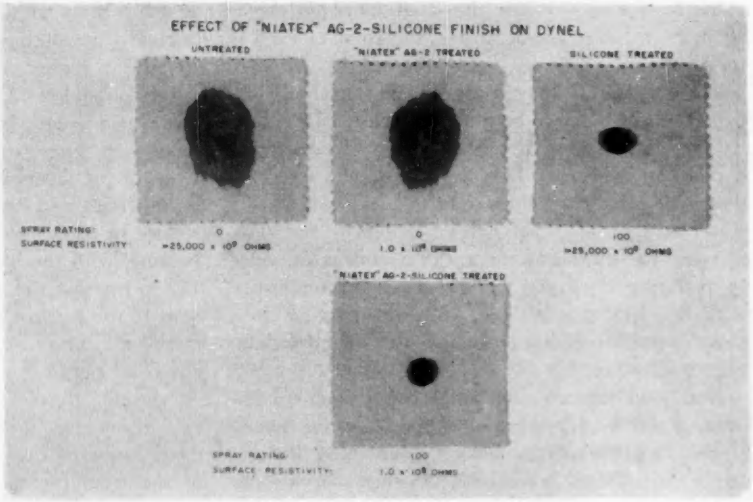

Fig. 9. Effect of treatment on water repellency and antistatic properties.

ment of these charges, but few will remain on a fabric or garment to provide protection after washing. The work of Hersh and Montgomery [3], which was done on the Union Carbide Fellowship at the Textile Research Institute, pointed up many of the problems and difficulties in obtaining reliable measurements.

Work at the Carbide laboratories on this difficult problem has resulted in the development of an antistatic finish, Niatex Antistatic AG-2, which has outstanding durability to laundering and dry cleaning on Dynel and many other fibers. This antistatic agent may be applied to fabrics by padding from an aqueous solution or to garments by spraying. In either case thorough drying is essential to obtain durability.

In Figure 8 are shown samples of untreated Dynel fabric and fabric treated with Niatex Antistatic AG-2 which have undergone 20 machine washes with All synthetic detergent. Both fabrics were subjected to conditions which would normally cause an electrostatic charge to build up; then they were held over a container of carbon black. Note that the untreated fabric shows a very high attraction and retention of carbon black, while the treated fabric did not attract dirt. Below each sample are shown surface resistivity measurements.

Niatex Antistatic AG-2 is compatible with a wide range of textile finishes. One of the more interesting and certainly surprising characteristics of this finish is its ability to emulsify certain silicone oils and thus to provide for Dynel and many other fibers finishes which have both durable water repellent and antistatic properties. Silicone emulsions are also compatible with Niatex Antistatic AG-2 and provide similar fabric finishes.

Figure 9 shows samples of Dynel treated with a mixture of Niatex Antistatic AG-2 and Dow Corning Silicone 104. The water repellency is indicated by the spray rating and by whether a drop of water spreads on the treated fabric, while the good antistatic properties are indicated by the surface resistivity of each sample.

Literature Cited

1. Brown, Alexander, TEXTILE RESEARCH JOURNAL **25**, 969 (1955).
2. Field, T. A., Jr. and Freeman, G. H., TEXTILE RESEARCH JOURNAL **21**, 531 (1951).
3. Hersh, S. P. and Montgomery, D. J., TEXTILE RESEARCH JOURNAL **22**, 805 (1952).
4. Rankin, W. W. and Newman, T. H., *Am. Dyestuff Reptr.* **45**, 213 (1956).
5. U.S. Patent 2,543,316.
6. U.S. Patents 2,743,991; 2,743,992; 2,743,993.

Verel—Eastman's Modified Acrylic Fiber[1]

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Introduction

In this symposium on acrylic fibers we are attempting to find the answers to three questions: "Why is the chemical industry investing so heavily in so many acrylic fibers?", "What are the distinguishing characteristics of the various acrylic fibers?", and "What special roles will these fibers play in the future textile economy?"

The answers to these questions must involve a consideration of the physical, chemical, and functional properties of the various acrylic fibers. In this paper, we shall discuss these properties of Verel, Eastman's new modified acrylic fiber.

Verel was announced in March, 1956, by Eastman Chemical Products, Inc., a subsidiary of Eastman Kodak Company, and is being produced by Eastman Chemical Product's corporate associate, Tennessee Eastman Company, which developed the fiber. New production facilities representing a multimillion dollar investment are nearing completion for its manufacture.

Verel is available in three types insofar as shrinkage characteristics are concerned: Regular, a stabilized, low-shrink yarn; Type I, a medium-shrink yarn; and Type II, a high-shrink yarn. It is available in two types insofar as flame resistance is concerned; these are designated as Verel and Verel FR.

This fiber possesses the good physical characteristics of other well-known acrylic fibers as well as certain desirable properties usually associated with the natural fibers. The most important of these properties are remarkable whiteness; soft, kind hand; ease of dyeing; good flame resistance; controllable shrinkage; and relatively high moisture regain. It has outstanding value in selected fields of use.

[1] Presented at the annual meeting of the Textile Research Institute, March 14, 1957.

We shall consider first those properties and applications which Verel has in common with other acrylic fibers. The major portion of the discussion, however, will be devoted to the specific properties that distinguish it from other acrylic fibers.

Physical Properties

General

X-ray diffraction patterns of Verel show that it is a well-oriented, slightly crystalline fiber with peanut-shaped cross sections, as shown in Figure 1. Its refractive index is 1.538; Verel exhibits practically no birefringence. The specific gravity is 1.37, a value close to that of acetate, rayon, and wool. Moisture regain is 3.5–4.0% at 70° F. and 65% relative humidity.

Tensile

Verel is a strong, tough fiber, and in this respect is similar to other acrylic fibers. It has a tenacity of 2.5–2.8 g./den., an elongation of 33–35%, and a toughness index of 0.46 g.-cm./den.-cm. Figure 2 is a typical stress–strain curve. The fiber has a compliance ratio of 1.0, a modulus of 40 g./den., an average stiffness of 8.0, and a yield point of about 0.8–1.0 g./den. Therefore it is softer than most of the acrylic fibers and is quite similar to wool.

Elastic

Verel is a fiber of medium elasticity, and in this respect is comparable to other acrylic fibers. The elastic behavior is shown in Figure 3. At 2% elongation, it has a work recovery of 78% and a length recovery of 79%. At 5% elongation, it has a work recovery of 41% and a length recovery of 63%. At 10% elongation, it has a work recovery of 20% and a length recovery of 38%.

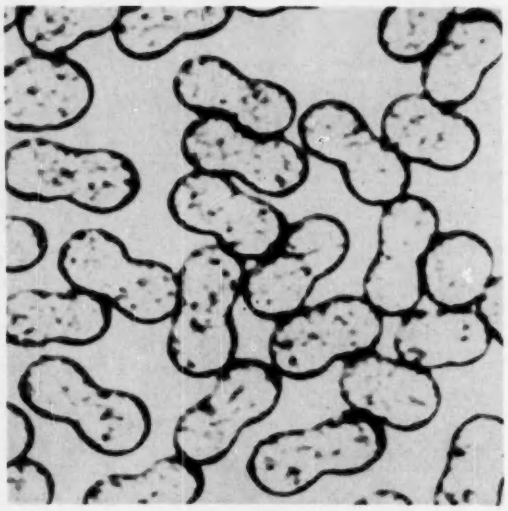

Fig. 1. Photomicrograph showing cross sections of Verel fibers.

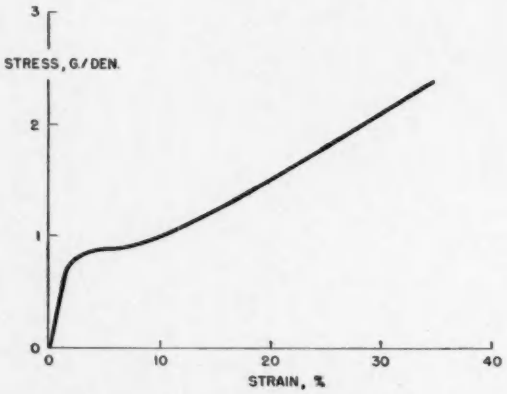

Fig. 2. Stress-strain curve for Verel.

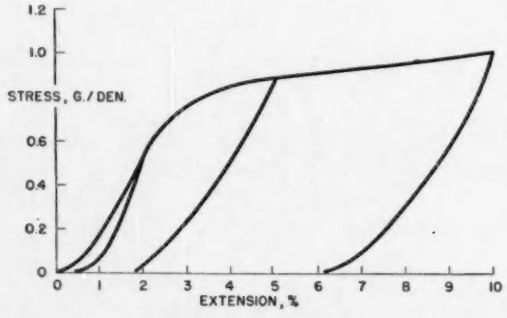

Fig. 3. Hysteresis of Verel at 2, 5, and 10% extension.

This recovery is instantaneous; much higher values are obtained when the fiber is given more time in which to recover. Verel has an elastic recovery of 88% at 4% elongation and of 70% at 10% elongation when a recovery time of 1 min. is allowed.

Thermal

Verel has no melating point; however, it is thermoplastic, and a maximum safe ironing temperature of 300° F. is recommended. Shrinkage of the fiber in boiling water is 0–2%. At 130–150° C., the fiber properties begin to change rapidly; the tenacity and modulus decrease and the elongation increases.

The effect of hot water on Type I and Type II Verel is shown in Figure 4. Type I shrinks 9–11% in boiling water, and Type II shrinks 19–23%.

In Figure 5 the effect of dry heat on these fibers is shown. Type I shrinks 15–17% at 150° C., whereas Type II shrinks 25–28%.

The fiber can stand exposure to moderate temperatures for a considerable time without loss in physical properties; samples were unchanged in strength or elongation after being heated at 110° C. for 128 hr.

Electrical

The dielectric strength of Verel exceeds 1500 volts/mil (measured on film). The ability to maintain its physical characteristics at extremely low temperatures and its flame resistance should make Verel useful in electrical applications.

Weathering

Verel has good weathering characteristics; it is better in this respect than any of the natural fibers and most of the synthetic fibers, including viscose, acetate, the polyamides, and the polyesters. The effect of outdoor weathering on tenacity and elongation is shown in Figure 6. Acetate, cotton, and wool were used as controls. After 50 weeks' exposure, Verel still had good strength and elongation, whereas the acetate and wool fibers were completely ruined and the cotton was too brittle and weak to be usable.

Chemical

Verel possesses a high degree of resistance to a wide range of chemicals. It has good resistance to

mineral acids, alkalies, oxidizing agents, all dry cleaning solvents, and most common organic solvents.

Biological

Verel is highly resistant to attack by mildew and other microorganisms. It was unaffected after being buried for 12 weeks in moist (30% water), biologically active river loam at 25° C.; cotton decomposed after 6–7 days in the same test. Extensive tests have shown that Verel is nonallergenic.

Dyeing and Finishing Properties

The preparation of Verel for dyeing follows the same general pattern as with other man-made fibers. Desizing with an enzyme in the case of insoluble starches or with warm water in the case of soluble sizing materials is necessary to insure even dyeing. Scouring or boiling off in a neutral bath with a detergent is normally sufficient for the removal of dirt and lubricants acquired in earlier processing. If scouring in an alkaline bath becomes necessary, a mild alkali such as disodium phosphate or tetrasodium pyrophosphate should be used.

Verel, as produced, is quite white and does not normally require bleaching. If, in some instances, it becomes necessary to whiten the fiber further, use of sodium chlorite with oxalic or formic acid is recommended. Peroxide or chlorine bleaches are not recommended for Verel alone or in blends, because the result is discoloration rather than bleaching.

There are three classes of dyes that are considered the most effective. They are the neutral-dyeing premetalized dyes; the dispersed, or acetate, dyes; and the basic, or cationic, dyes. The neutral-dyeing premetalized dyes offer the best all-around light and wash fastness. The dispersed, or acetate, dyes may be used to obtain brighter shades, in some cases, than are possible with the premetalized dyes, but color fastness generally is somewhat less. The basic, or cationic, dyes are generally used for specialty fabrics when brighter shades are required than can be obtained with either the premetalized or acetate dyes. Normal dyeing methods are employed. Operating temperatures of 180–205° F. are sufficient to exhaust these dyes completely over the full range of shades. In dyeing heavy shades, a special assistant is used to obtain a complete exhaustion of the dye on the fiber or fabric.

Blends of Verel with other textile fibers may be dyed using almost any desired combination to obtain union shades, color and color, or color and white

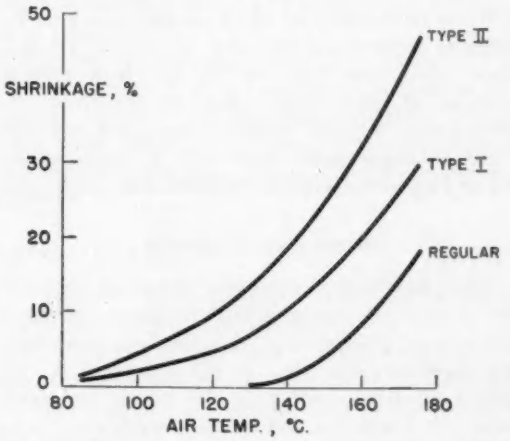

Fig. 5. Effect of air temperature on shrinkage of Verel.

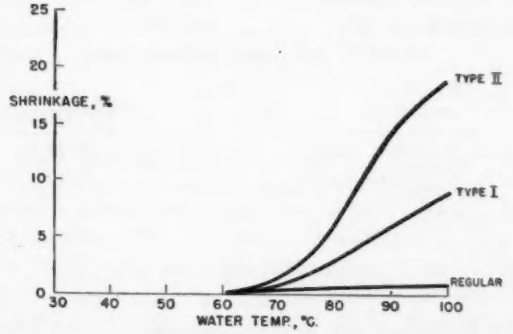

Fig. 4. Effect of water temperature on shrinkage of Verel.

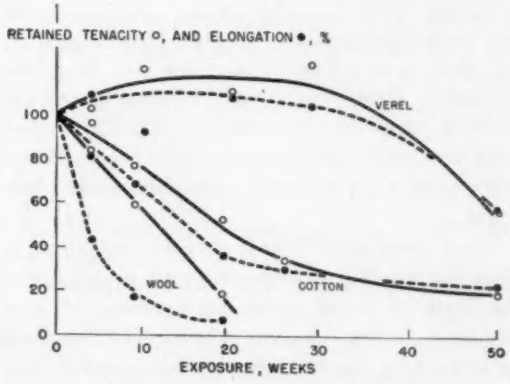

Fig. 6. Weather resistance of Verel.

effects. Blends with rayon or cotton offer a multitude of possibilities, since Verel may be dyed with either premetalized or acetate dyes, and the rayon or cotton may be dyed with direct dyes. With such combinations there is little, if any, cross-staining. Blends with wool offer a very outstanding feature in that selected premetalized dyes will dye union shades on the two fibers without resorting to extended dyeing periods or the transfer of dye from one fiber to the other. Other commonly used textile fibers may be processed in blends with Verel to obtain a great many color combinations.

In finishing fabrics containing Verel, as in the case of other hydrophobic fibers, certain precautions should be observed. Thermosetting or reactive resins commonly used on cellulosic fibers are not effective on Verel, since these materials remain on the fiber surfaces. The result is an undesirable stiffness or boardiness, which impairs many of the inherent properties of the fiber. Blends of cellulosic fibers and Verel may be finished with reduced amounts of these resins when, by selectivity, the resin reacts with the cellulosic fibers but not with Verel. Temperatures above 300° F. cause stiffening and should be avoided in all finishing operations.

Processing Properties

Verel has been satisfactorily processed on all of the systems of spun yarn manufacturing; no major changes or modifications in processing procedures are required. The fiber, as shipped, requires very little mechanical opening on the pickers and card; therefore, low beater and lickerin speeds are recommended. Use of a fancy roll on the flat card is also recommended; it is not needed when carding blends containing 50% or more viscose. Normal speeds and production rates are maintained for manufacturing spun yarns. Evaluation of this fiber in various customers' plants has shown it to process well and produce yarns of good quality. Verel also lends itself very well to blending with other fibers. Blends with wool, viscose, cotton, and Estron have been made.

Since Verel staple fiber is available in both stabilized and unstabilized forms, mills not equipped with the Turbo or Pacific system can produce their own shrinkable or bulking yarn. Unstabilized fiber, such as is used for the backing yarn for imitation furs, does not process quite as well as regular, or stabilized, fiber. This means that the unstabilized fiber does not spin quite as fine as the stabilized fiber.

Verel can be tinted for identification. However, it has been found that most mills prefer processing the acrylic fibers in the natural or untinted state. Should tinting be necessary, an application of a tinting solution at the picker will give satisfactory results.

No special techniques are needed in winding, warping, and weaving. However, warps containing all or a high percentage of Verel should not be stretched unduly in slashing, unless a shrinking or bulking action is desired in the finished fabric. The size box temperature should be 140–150° F. in order to control the stretch during slashing within a range of 5–10%. Any size now being used on other acrylic fibers will be satisfactory on Verel. The sizes that we have found satisfactory consist of a converted starch, Elvanol or Stymer R, a wax, and a preservative such as Dowicide A. The actual composition of the size will depend on the mill and the construction being woven. Normal drying temperatures are suitable.

A few of the practical yarn counts that can be spun from Verel are shown in Table I. In the case of wool blends, almost any count desired can be spun with good strength and quality.

Textile Properties

Verel, like the other acrylic fibers, has a number of functional properties that make it of particular value to the textile trade. Among the most important of these are

1. Good tensile strength.
2. High resistance to shrinkage.
3. Excellent resistance to chemicals.
4. High resistance to moths and mildew.

TABLE I. Spin Limits for Verel Staple

Filament size, den./fil.	Filament length, in.	Spin count
2	1.5	40/1
2 (50%) / 3 (50%)	1.5	31/1
3	1.5	27/1
3	2	30/1
3	2.5	34/1
2*	1.5	68/1
3**	2	34/1

* 50/50 blend with combed Pima cotton.
** 50/50 blend with 1.5 den./fil., 2-in. cut viscose.

5. Excellent weather resistance.
6. No allergenic activity.
7. Remarkable whiteness.
8. Low pilling tendency.
9. Good press and shape retention.
10. Good wrinkle recovery.

Verel, by virtue of its unique chemical composition, possesses a number of physical characteristics which distinguish it from the other acrylic fibers; the remainder of this article will be devoted to a discussion of these properties.

Softness

Verel has an unusually soft, kind hand—a quality which connotes a feeling of luxury coupled with warmth. Its softness, as well as its other properties, makes Verel of considerable interest for making pile-type fabrics. Such fabrics include man-made furs of both knitted and woven types; trim for cuffs, collars, boots, and shoes; and a wide range of liner fabrics. Verel is used in both the face yarn and the backing yarn of pile-type fabrics.

In knit goods, the ability of Verel to retain its soft hand after repeated laundering and drying recommends it for use in blends with cotton for sport shirts, undershirts, and children's sleepers. In such end uses, the determination of fiber to be used and the percentage in the blend are greatly affected by economic factors. Twenty-five per cent blends with cotton are well within the economic requirements of the market and, at the same time, utilize the properties of Verel which contribute to hand, dimensional stability, and color.

In men's wear, particularly flannels, Verel in proper blends with medium wools will allow higher spinning limits. This counteracts the wiriness of the wool and produces a fabric with an improved hand.

Ease of Dyeing

Verel is readily dyeable. Ability of this fiber to be dyed at normal operating temperatures rather than at a boil or under pressure enables any dyer to process Verel fiber or fabrics without special equipment. Its color fastness is outstanding in the acrylic family of fibers. The whiteness of the fiber as it is produced allows a full range of shades from clear, bright pastels to blacks and navies.

Controllable Shrinkage

The availability of Verel in both stabilized and unstabilized types provides the designer and technician with the means to build fabrics having any desired bulk, hand, porosity, or surface characteristics. These types of staple can be used to produce high-bulk fabrics of any desired blend using conventional equipment.

Additional work must be done to produce a fine quality sweater from 100% Verel fiber; however, Verel lends itself very well to use in sweaters and other knitted outerwear. The proper blending of stabilized and unstabilized Verel fibers results in a knitted fabric possessing a very kind hand, excellent dimensional stability, and a low pilling tendency.

The proper selection of blends with other fibers makes it possible to build pile fabrics of varied height, simulating the guard and body hair structure of an animal fur.

The use of shrinkable yarns in woven goods is well illustrated by the so-called three-dimensional fabrics. A wide range of blister, or pucker, is possible; thus, fabrics for the varied requirements of the decorative (drapery and upholstery) trades are provided.

Moisture Regain

A moisture regain of approximately 3.75% places Verel well up the scale in comparison with other synthetic fibers. This property assists in mechanical processing and contributes to comfort by providing freedom from the clamminess associated with many low-regain fibers.

Although higher moisture regain adds to comfort in all end uses, this property is particularly valuable when Verel is blended with wool and cotton for socks. Tests show that blends containing 30–50% of Verel with wool or cotton give good dimensional stability, a comfortable feeling, and little tendency toward pilling.

Flame Resistance

Verel is a flame-resistant fiber. Type FR Verel is extremely flame resistant.

There are numerous tests designed for the testing of the flame resistance of fabrics. The five most stringent of these are as follows: Underwriters' Laboratories, Inc., Federal Specification CCCT-191B, State of California, City of Boston, and New

York City tests. These involve the ignition of a 2- × 10-in. or 2- × 12-in. strip held vertically over a luminous flame for 12 sec. The sample is clamped in place in most of these tests so that the fabric can not shrink away from the flame; however, the New York City test does not require that the sample be clamped. The more drastic tests specify that the average afterflame be 2 sec. or less for 10 samples. Also, the length of char shall not exceed 4½ in. for material which weighs 6–10 oz./sq. yd. The New York City test requires that there be no flashing, an average afterflame not exceeding 3 sec., and an average afterglow not exceeding 20 sec. (outside the charred area). Type FR passes all these fire laws and codes. By virtue of its inherent flame resistance, Verel offers a built-in safety factor which should be of value in both apparel and decorative fabrics.

Summary

Verel has the following properties in common with other acrylic fibers: whiteness, good chemical resistance, excellent moth and mildew resistance, good strength, and excellent weatherability.

By virtue of its unique chemical composition, Verel possesses a number of physical characteristics which distinguish it from other acrylic fibers. Among the most important of these properties are

1. Soft, kind hand—which imparts a luxurious feel to fabrics.
2. Ease of dyeing—which allows for simplified processing.
3. Flame resistance—which provides a considerable safety advantage.
4. Controllable shrinkage—which allows for the easy production of bulk yarns and pile-type fabrics.
5. High moisture regain—which contributes added comfort.

The success of any fiber is determined principally by its properties and the economics of its production. Its performance–cost ratio must be more favorable than that of other fibers in enough applications to support an economic volume of production. Verel, which is well qualified in this respect, is developing a substantial market and is expected to play an important role in the textile economy of the future.

Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Fiber Replication for Light Microscopy

Tennessee Eastman Company
Research Laboratories
Kingsport, Tennessee
April 11, 1957

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In the study of fiber surfaces, it is frequently desirable to examine relatively large areas in the magnification range of 1500 to 3000. Because of physical limitations, the electron microscope cannot satisfactorily cover large areas, and light microscopy is generally limited because the field lacks flatness and contrast. It is only natural that the procedures of electron microscopy should be employed with those of light microscopy to develop a method by which adequate areas of fibers might be examined with good contrast.

Shadow casting of fiber surfaces has been used by many researchers, but little work has been reported in which the shadow-replicas were removed and flattened for observation. By using this method, one is able to improve contrast and, therefore, resolution and the flatness of field. It is then possible to examine large surface areas as if they were relatively flat. The purpose of this letter is to describe the method which we use in our laboratories for surface examination.

The fibers to be studied are cut approximately 2 in. long, cleaned of lubricants by washing in ether, and then placed lengthwise on a glass slide. On one end of the slide, the fibers are spread out with tweezers so that there is no overlapping, and Scotch tape is pressed down over the ends of the fibers to secure them in a flat, parallel position. A soft camel's hair brush cut flat on the end may then be brushed along the fiber group to cause separation and parallelism. When the fibers have been made parallel and flat on the slide, the free ends may be taped to the slide to hold the fibers in position. Care must be exercised that no two fibers cross, as this will cause replica breakage.

The slide and fibers are then placed in a vacuum coater, and aluminum is evaporated for shadow casting. We believe that it is advantageous to place the filament so that shadows are cast along the fiber axis and at approximately a 3 to 1 angle. The angle will vary slightly from one end of the fibers to the other and will be slightly away from the axis on the sides of the fibers. The aluminum coating should be about 1000Å thick to produce good reflectivity and strength, but should be sufficiently flexible to flatten well in later operations.

A second glass slide is coated with a smooth, thin film of D-galactose. This film is easily obtained by spreading 2 or 3 drops of a 5% solution of galactose in water over approximately 1 sq. in., drying on a hot plate, and cooling to room temperature. The film is rendered tacky by exhaling moist air from the mouth across it. This slide, coated side down, is then placed perpendicular to the aluminized fibers and pressed onto them. The Scotch tape holding the fibers down should not be between the slides. With the slides held firmly together, the fibers may be cut

with a razor blade where they protrude from beneath the coated slide. The latter may then be lifted with the fibers imbedded in the damp galactose, which may be hardened by gentle heating or vacuum drying. This hardening firmly imbeds the fibers with the replicas next to the galactose.

The next step is determined by the type of fibers with which one is working. The best procedure is to dissolve the fibers in a solvent not affecting the galactose. For example, cellulose acetate fibers may be dissolved in dry acetone. However, insoluble fibers may be mechanically removed with tweezers, leaving the replicas imbedded in the galactose.

Once the fibers have been completely removed, the replicas are floated onto water by slowly immersing the slide at an angle into a dish of water and allowing the sugar to dissolve. With careful manipulation, long strings of fiber replicas can be obtained. When a clean slide is immersed in the water and slowly raised under the replicas, starting at one end and allowing the water to drain off as it is raised, they will remain intact and quite flat. Since the replicas dry in contact with the glass, they are very sturdy and can be used directly with oil immersion. Examination may be made with transmitted light or metallographic vertical illumination.

VIRGIL PECK

Aluminum Replicas of Fiber Surfaces for Light Microscopy

E. I. du Pont de Nemours and Company, Inc.
Textile Fibers Department
Wilmington, Delaware
June 10, 1957

To the Editor

TEXTILE RESEARCH JOURNAL

Dear Sir:

Although examination in the electron microscope of replicas of fiber surfaces has given valuable information, questions concerning gross structures and those irregularities which occur only periodically along the fiber were essentially left unanswered, since, at best, only small total areas could be examined. Examination of these fibers in the light microscope gave some additional information, but, in general, proved inadequate because (1) internal structures interfere with the resolution of surface characteristics, (2) the curvature of the fiber and the short depth of focus of the optical system permits only a small area to be in focus at any one time. Evaporation of an aluminum film onto the fiber surface eliminated the first problem, but the second remained. To circumvent this, aluminum replicas were prepared; however, there was some difficulty in stripping the fibers from the replicas and flattening the replicas themselves. The procedure used in this laboratory has solved these last two problems to the extent that satisfactory replicas for light microscopy can be prepared on a routine basis. To develop image contrast in these replicas, two techniques are available. First, one may shadow with aluminum at an oblique angle, as done in electron microscopy, thus showing detail by different thicknesses of metal, which produce varying amounts of light absorption or reflection. Second, one may evaporate from a relatively broad source normal to the fiber surface and produce shadows by using oblique illumination. In the work described here, we chose the latter method because it produced a stronger replica that allowed us to vary the shadow direction at will. It should be pointed out, however, that this technique is more difficult to set up because any error in the position of illumination will produce a loss of contrast.

To prepare samples for replication, single filaments are mounted across $\frac{1}{4}$ in. wide glass strips cemented to a microscope slide. The width of the space between the glass strips is slightly smaller than the width of a microscope slide. A film of aluminum is then evaporated at normal incidence onto the fiber surfaces. A microscope slide covered with a thin layer of 3% polyacrylic acid in water is placed over the filaments with its edges resting on the glass strips of the jig; the solution is allowed to dry. This slide is then separated from the original mount and any adhering filaments pulled off. A film

of polystyrene is cast from a carbon tetrachloride solution over the replicas and polyacrylic acid film. When thoroughly dry, the slide is immersed in hot water and the polystyrene film is picked up on a microscope slide and dried; the polystyrene is washed off with carbon tetrachloride. The dry slide is again wet with a solution of polyacrylic acid and dried. This last wetting helps flatten the replica.

A type of illumination referred to as oblique dark field produces the best contrast and definition of structure. An adjustable dark field condenser is so displaced that the light is deflected off the edge of the objective onto the sample, then reflected back by the sample to the objective. A 90× oil immersion apochromatic objective and a compensated 10× eyepiece are used.

The above technique may also be used in replicating films and other surfaces by slight variations in the basic procedure.

ANGELA W. FERGUSON
ROBERT G. SCOTT

Standard Reference Fabrics for Soil Burial and Weathering Tests

Corps of Engineers, US Army
Engineer Research and Development Laboratories
Fort Belvoir, Va.

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

The need for a standard reference fabric that could be used by all laboratories in studying the performances of fungicides in soil burial, weathering, and other tests has long been recognized. To resolve this need, representatives from various governmental and commercial agencies met at the National Academy of Sciences in Washington, D.C. to discuss the possibility of adopting such a fabric for interlaboratory use.

At this meeting it was proposed that two standard fabrics be adopted: (1) an untreated cotton duck, made to exacting specifications to ensure uniformity, and (2) the same duck treated with an acceptable fungicide as a treated control standard.

The preparation of uniform 1-in. raveled specimens from these standard fabrics would be facilitated by the use of colored warp yarns so placed that they would outline exactly 1 in. of the warp.[1] Raveling to these colored yarns would give replicate 1-in. specimens without the necessity of measuring.

[1] TEXTILE RESEARCH JOURNAL **26**, 621–622 (August 1956).

The use of these standard reference fabrics would provide:

a. A means of determining the change in activity of a given soil bed from one test period to another.

b. A means of comparing directly the effectiveness of fungicides which were tested at different times.

c. A means of comparing the activity of soil beds used by different agencies.

d. A means of determining the severity of different weathering periods.

e. A means of taking into account shrinkage or expansion during treatment. The number of threads bounded by the two-colored yarns will always remain constant.

The cost of these fabrics would be only slightly higher than that of ordinary fabric of the same weight. It is planned to have woven and treated at one time the estimated needs of cooperating laboratories for a period of three years. In the interest of standardizing fungicide test procedures, all interested laboratories are offered the opportunity of including their needs in this master order. Information can be obtained from Samuel Shapiro, Fungus Control Section, Materials Branch, U.S. Army Engineer Research and Development Laboratories, Fort Belvoir, Virginia, or Patrick J. Hannan, Code 6120, Naval Research Laboratory, Washington 25, D.C.

SAMUEL SHAPIRO

Book Reviews

Wool Research, Vol. 2. Physical Properties of Wool Fibres and Fabrics. Wool Industries Research Association, Leeds, England, 1955. 234 pages. Price 30 shillings.

Reviewed by Joseph H. Dusenbury, Textile Research Institute, Princeton, N.J.

This volume is the fourth issued in the "Wool Research" series published by W.I.R.A. It should be noted that these books are not coming forth in numerical sequence. Thus, Volume 4, "Carding," appeared with a 1948 copyright date; Volume 6 on "Drawing and Spinning" appeared in 1949; and prior to the present volume under review, Volume 3, "Testing and Control in the Wool Industry," was issued in 1955.

The following topics are treated in this volume: relative humidity, regain, and swelling; absorption and desorption of water; warmth of clothing; water repellency; elastic and plastic properties; electrical properties; friction; and color and optical properties of fabrics. In comparison with its predecessors, which appear to have been put together as scissors-and-paste assemblies of earlier W.I.R.A. published reports, the present volume has only two chapters so prepared. The remaining chapters represent a capable rewriting of earlier results, and in some instances there has been a reinterpretation of these results. This has resulted in some new deductions from previous data in several cases, and the reader is indebted to F. L. Warburton for this work. In fact, this single authorship of most of the chapters has served to enhance the readability of the entire volume.

It is stated in the Preface by Dr. Cassie: "The book has been written with an eye to the practical needs of the wool textile industry and because of this no mention is made of those long-term research projects of the Physics Department that are still in the academic stage." The reader is cautioned, however, that the application of what is in this volume to practical needs probably will require the services of someone trained in physics or physical chemistry. Those without such a background may find the reading rather heavy going at times. The reader of scientific bent will be pleased with the serious attempts made to attack such complex problems as the warmth of clothing and water repellency. The latter topic is covered in Chapter 5, which is essentially a reprint of the two classic papers on the subject written by Cassie and Baxter over ten years ago. The other chapter not written by F. L. Warburton is No. 8, "Friction," by B. Lincoln. While the chapter by Cassie and Baxter is timely, mainly because little important work has been done since their efforts, the chapter by Lincoln has the same characteristics because of its covering the field up through 1954. In the friction chapter, Lincoln has considered work other than that done under W.I.R.A. sponsorship; the same cannot be said for much of the rest of the book. This treatment of non-W.I.R.A. work gives this chapter and other portions of the volume where it occurs a more catholic flavor, but it must be pointed out that it is the announced intention of this series to consider only W.I.R.A. papers. This intention has been carried out fully in the chapter on elastic and plastic properties, and the reader should consider this restriction in going through this and other chapters.

The format of the book is attractive, and it is a comfort, at least to this reviewer, to realize that in these days it is possible to put out such a volume for the relatively low price indicated. With the cautions mentioned earlier for the prospective reader kept in mind, the book is recommended as an excellent source of information on the physical properties of wool. The knowledge presented in it should be valuable to scientists both with and without prior experience in the field.